ELECTROFORMING PROCEDURES FOR SHELLS OF MOULDS AND DIES FOR SUBSEQUENT SIMULTANEOUS SPRAY PEENING

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DECLARATION

I hereby declare that this thesis has been composed by myself, that it has not been accepted in any previous application for a degree, that the work of which it is a record has been carried out by myself, and that all sources of information have been specifically acknowledged.

Xiang-Kang Chen

ABSTRACT

With the development of automotive and toy industries, more and more plastics injection dies and moulds are required. Many researchers attempt to find new processes, which can reduce manufacturing costs and delivery time and obtain dimensional accuracy and high quality surfaces for dies and moulds. This thesis describes the development of a novel hybrid process for the manufacture of plastics injection moulds.

Compared with conventional procedures, the hybrid process can produce high quality plastics injection moulds at low cost and with short lead times. The hybrid process consists of two stages - electroforming and simultaneous spray peening. This work undertakes research and development of electroforming aspects of the hybrid process. All techniques and theories will be discussed in this thesis.

The feasibility of using electroformed shells to make the working surface of plastics injection moulds was investigated. The important mechanical properties of a nickel shell, such as hardness, tensile strength, ductility and internal stress, were researched and improved by using various operating conditions and adding some agents. The pulse plating power supply was designed to improve the mechanical properties of the nickel shell. The influence of pulse plating parameters on electroforming shell accuracy was analysed. Mandrel materials were also investigated. The accuracy of reproduction by electroforming was measured and analysed. The interface between the nickel shell and spray metal was analysed by scanning electron-microscope and the bond strength measured using a direct tensile testing method.

Finally, two injection moulds were made up and tested under two conditions, plastics injection and high pressure casting of aluminium. The results show that the moulds made by the hybrid process can be used successfully to produce plastics components, but can not be used for high pressure of casting aluminium. This application of the hybrid process has shown promise in the automotive and toy industries.

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ABBREVIATION

CAD ------	Computer Aided Design
CLA -------	Centre Line Average
CNC -------	Computer Numerical Control
CPU --------	Central Processing Unit
D.C. -------	Direct Current
DTI --------	Danish Technological Institute
ECAM ------	Electro-Chemical Arc Machining
ECM -------	Electrochemical Machining
EDM -------	Electrodischarge Machining
MUST ------	Manufacture tools and dies Using Spray forming Techniques
NC -------	Numerical Control
PVC ------	Polyvinyl Chloride
Ra --------	Roughness of Average
Rc --------	Rockwell Hardness
SDL -------	Sprayforming Developments Limited
SLA -------	Stereolithography
SSP -------	Simultaneous Spray Peening
Vc --------	Velocity of Cutting
VHN ------	Vickers Hardness Number
I_a ---------	Average Current Density

PART ONE - INTRODUCTION AND REVIEW

CHAPTER 1

INTRODUCTION

1-1. OBJECTIVES AND ORGANISATION OF THESIS

1-1-1. Objectives of the thesis

With the development of die and mould manufacturing industries, more and more companies try to find new processes, which can reduce manufacturing costs and lead times, and obtain dimensional accuracy and high quality surfaces for dies and moulds. The purpose of the present thesis is to develop a new hybrid process, which can satisfy the above requirements. In order to gain more insight into this new process, the following topics will be investigated:

1. the mechanical properties for the electroformed shell produced by both D.C.* and pulse power supply.

2. the effect of plating parameters on the internal stress.

* Please refer to Abbreviation section on page xi.

3. the influence of pulse plating on electroformed shell dimensional accuracy.

4. materials for the mandrel.

5. bond strength between the electroformed shell and SSP coating.

Real dies and moulds for high pressure casting of aluminium and plastics injection were manufactured and tested.

1-1-2. Thesis Organisation

This thesis is composed of three parts. In part one, there are two chapters; chapter 1 - "Introduction" and chapter 2 - "Review of Electroforming". Part two contains experimental procedures and results, including chapter 3 - "Mechanical Properties of Electroformed Nickel Alloys" , chapter 4 - "Pulse Plating", chapter 5 - "Formers and Patterns" and chapter 6 - "Interfacial Analysis and Mould Tool Development". Part three has only chapter 7- "Discussions and Conclusions". Each chapter is summarised as follows:

Chapter 1: Die and mould manufacturing is reviewed. Several processes to make dies and moulds are introduced. The new hybrid process is described.

Chapter 2: The history and principle of electroforming are reviewed. The advantages and limitations of electroforming are discussed. Some applications of electroforming are introduced. The basic experimental apparatus of electroforming is also mentioned.

Chapter 3: The mechanical properties of electroformed nickel alloys are investigated in this chapter. The hardness, tensile strength and elongation of the electroformed shell are measured. A method of measuring internal stress is designed and the effect of deposition variables on internal stress is studied.

Chapter 4: A pulse power supply is designed to investigate the influence of pulse plating parameters on hardness, internal stress and accuracy of the electroformed shell. The characteristics of pulse plating are also discussed.

Chapter 5: The different mandrel materials, conductive and non-conductive, are investigated. Considering cost and ease of removal of the mandrel, the best materials are chosen. The influence of current and metal distribution on the nickel shell is analysed. The throwing power is also studied in this chapter. Finally, the accuracy of reproduction by electroforming is measured and analysed.

Chapter 6: Various procedures to improve the strength of the bond layers between the electroformed shell and the sprayed metal are investigated. A method of measuring the bond strength is introduced. The bonding strengths with various etched surfaces are measured. The interface with different treatment is analysed. Two injection moulds are manufactured and tested under two conditions, plastics injection and high pressure die casting of aluminium. The test results are shown in this chapter.

Chapter 7: This chapter gives the discussions and conclusions with recommendations for further work.

Appendix: The published material "Tools Made by Electroforming and Simultaneous Spray Peening" is given.

1-2. REVIEW OF DIE AND MOULD MANUFACTURING

Dies and moulds enable manufacturing industry to produce, economically, large numbers of discrete components with precise dimensions. Modern die and mould manufacturing processes include most aspects of manufacturing from design to finished product. The process covers part design, die design, process modelling, rapid prototype production, control of dimensional and surface quality by various conventional and modern processes including advanced mechanical, electrical, and electrochemical machining methods.

A large percentage of parts is manufactured by using a near net shape process, such as forging, die casting, stamping or injection moulding. These processes utilize dies and moulds to impart the near net shape on incoming raw material or a pre-formed workpiece. However, the geometry, tolerance, surface finish and strength requirements have to be considered during the manufacturing of dies and moulds. Intricate geometry with sculptured surfaces and relatively high material hardness make die and mould design and manufacture increasingly difficult. In addition, each process has its own special requirements for the die or mould characteristics. For example, hot forging and die casting dies are subject to thermal fatigue and erosive wear, while cold forging dies must sustain very high stresses, and must be resistant to mechanical low cycle fatigue; injection moulds must have excellent surfaces and be resistant to corrosion, indentation and wear.

In most countries the die and mould industry consists mainly of small companies, typically with fewer than 100 employees. The users of dies and moulds increasingly expect that:

1). manufacturing costs and lead times are reduced.

2). dimensional accuracy and overall quality are improved.

3). dies and moulds suitable for the production of short prototype series are available quickly for testing and evaluation purposes.

4) design changes can be easily accommodated.

As a result of these requirements, die makers are forced to improve communication with their customers, raise the training level of their employees, and utilize modern manufacturing technologies such as high speed machining, CNC milling machining, electrodischarge machining, abrasive processes and electrochemical machining.

The processing steps in die manufacturing may generally be divided into: die design, tool path generation, rough machining (of die block and / or EDM electrode), finish machining, polishing (including manual and automated polishing) and die try-out One researcher [74] surveyed the injection moulding industry in U.S.A. and reported that the total mould manufacturing time varied between 1,200 to 3,800 hours, depending on mould size, geometry and complexity. The time spent for each manufacturing step was in the range of: 15 - 24% for design, 14 - 17% for tool path generation, 8 - 16% for rough machining, 27 - 39% for finish machining, 13 - 23% for polishing, and 4 - 6% try-out.

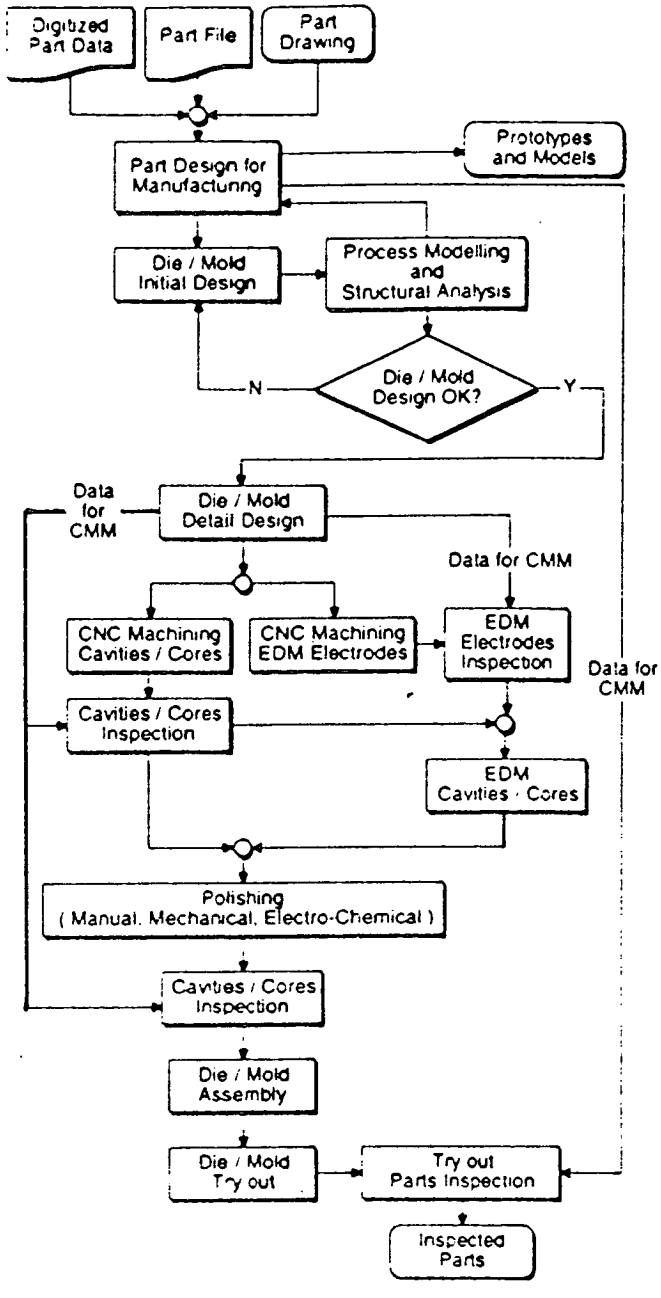


Figure 1.1. Information Flow and Processing Steps in Dies and Moulds Manufacturing [1]

Altan and Hilly [1] summarized the information in Figure 1.1 and demonstrated that die and mould manufacturing must be considered as a total system which includes:

i). part design for near net shape manufacturing;

ii). production of prototypes for visualization, testing and evaluation;

iii). process modelling for optimum mould design;

iv). CNC machining;

v). manufacturing of EDM electrodes;

vi). Electrodischarge Machining (EDM);

vii). surface conditioning and polishing;

viii). dimensional control of electrodes, die and near net shape formed product.

It is well know that the design activity takes only a small part, 5 - 15%, of the total production costs of a component. However, decisions made at the design stage have a profound effect upon its manufacturing and life cycle costs. In addition to satisfying functional requirements, for the selected near net shape process, the part design must consider the following four points:

a). geometric complications that affect productivity.

b). equipment and tooling requirements.

c). process capabilities such as size, geometry and production rate.

d). properties of the incoming material.

During the last decade, various processes have been developed for rapid production of plastic and ceramic models directly from a CAD model. These processes, generally considered to be "rapid prototyping" techniques, are classified under various names such as direct CAD manufacturing, three dimensional printing,[66] desk top manufacturing, instant manufacturing, solid free-form fabrication, layer manufacturing, material deposition manufacturing, or material accretion manufacturing. Also major efforts are devoted to the development of prototype expert systems in cold forging, sheet metal forming or injection moulding, which appear to find large industrial acceptance. In this last case, a commercial CAD system is used for designing standard as well as non-standard mould components.

As a result, there are several commercial programs that are routinely used, even by small companies, in die casting and injection moulding. The process simulation software available today not only allows the prediction of material flow, temperature, and solidification, but it also estimates the elastic recovery as well as residual stresses, microstructure, and in some instances even properties in near net shape parts.

1-2-1. Manufacturing Dies and Moulds by Conventional Mechanical Methods

Most dies and moulds consist predominantly of convex surfaces; thus they can be easily machined by 3-axis or 5-axis CNC mills. On the other hand, some cavities may have deep pockets and surfaces that may be difficult to reach and produce by milling machining. For this reason, sinker EDM is often selected to

finish machine cavities. Most die and mould manufacturers use 3-axis machining, although 5-axis milling offers evident advantages in machining large dies and moulds with convex surfaces. By using flat end mills while keeping the cutter axis orthogonal to the cavities surface, an improved surface finish is obtained. In addition, set-up requirements and machining time may be reduced up to 50% of that obtainable with 3-axis milling. [87]

Other researchers[41] pointed out that in CNC milling of dies and cavities the following major process steps and variables are considered:

A). NC path generation.

B). advanced machining techniques such as high speed milling and machining of heat-treated die steel.

C). tool materials and monitoring.

D). machine tool characteristics and controls.

In addition to these technological considerations, modern cost effective die manufacturing techniques require integrated planning of the roughing, finishing, and polishing steps.

Generally speaking, most dies and moulds need quite good accuracy and surface finish. It is very difficult to obtain these requirements by milling. Some highly advanced die and mould manufacturing companies seem to prefer to produce their dies with advanced CNC milling followed immediately by EDM techniques, i.e. using CNC millings for rough machining, then using EDM techniques to obtain a good surface finish.

With the development of advanced manufacturing technology, one of the new processes, high speed machining, appears in the die manufacturing field. Some researchers [70] claimed that all die and mould milling operations - rough, semi-finish and finish machining - can benefit from high speed machining technology, with high feed rates in the range of 15 m/min, high spindle speed (20,000 rpm or higher) and surface cutting speeds of 700 m/min. In die manufacturing the objectives of high speed machining are to: reduce benching (hand polishing) time, improve accuracy and surface quality, reduce lead time and finally, reduce cost by unattended machining.

For high speed machining it is necessary to have special CNC control, a rigid and dynamically balanced spindle and machine tool structure, and specialized milling cutters. The CNC control must have a high speed data processing capability, usually, in addition to a standard 32 bit CPU is used to facilitate rapid processing of part program data. For a line segment of 0.3 mm, and feed rates of 4,000 - 5,800 mm/min, block processing times required are in the range of 4 to 5 ms. In order to feed a control that processes NC data in 4 ms, it is necessary to have high transmission speed. Also high speed milling machines are available to cut hardened steel, for example H13 at Rc 50, using a surface cutting speed of 300 m/min, coolant delivery at 1,000 psi through the spindle, a 10 mm diameter tool at 12,000 rpm and a feed rate of 2,000 m/min.[74] Further special milling machines are available with up to 15 m/min. feed rate, 95,000 rpm spindle speed, and a maximum positioning accuracy of 5 μm.

High speed machining processes can achieve very high metal removal rates, but there are critical requirements for the high speed cutting tools. Four aspects have to be considered: tool material and coating; cutting edge geometry; tool

design; and interface between tool and spindle. Advanced tool materials are able to machine hard steel at high metal removal rates. For example, the best performance of tungsten carbide in finish milling of a hardened hot work die steel is achieved by increasing the cutting speed from the conventional Vc = 140 m/min. up to 500 m/min. At this high speed, the tool life decreases only slightly and the inserts can cut tool steels economically at speeds up to 900 m/min. Coating of carbides with titanium nitride further increases tool life in hard machining at high speed.

At present, tool monitoring on a real time basis depends on the control of feed rate based on spindle loading, and automatic changing of worn out tools without operator intervention. Thus, totally unattended milling becomes a realistic possibility.

1-2-2. Die Sinker EDM Process.

It was well known that Electrodischarge Machining (EDM) came to metal workshops in the early 1950s and has become a key process for producing small and medium size precision dies and moulds. [94] With the developing of new EDM technology, it plays a major role in manufacturing dies and moulds with sculptured surface.

The die sinker EDM is best suited for machining deep and narrow cavities in hard materials. Most die and mould companies have EDM equipment, which is applied to a wide range of parts. However, the process is relatively slow compared to milling. In addition, the use of conventional dielectric fluids causes environmental concerns, and a potential fire hazard. Many researches,[27,33,49,50,51]]

in recent years, have been devoted to improving the machining conditions including the use of non-flammable dielectric fluids, such as water-based EDM dielectrics, orbital EDM to increase machining accuracy, and the reduction and control of electrode wear.

An increasing number of modern CNC EDM machines have an orbital electrode system. The EDM electrode can be rotated and a relative motion between the electrode and the workpiece can provide very complex paths. Therefore, it is possible to use same electrode for roughing and finishing. During orbital EDM, electrode wear is reduced, leading to improved cavity tolerances. Usually, in planning the orbiting EDM tool path, it is necessary to consider electrode erosion because the electrode is constantly changing its orientation with respect to the workpiece and as a result the working area changes as well. On the other hand, orbiting gives more efficient flushing of the dielectric, owing to agitation caused by moving.[53] It was found that although orbiting aids in flushing, removal of debris from the gap still remains a primary concern because gap contamination through eroded debris influences ignition as well as discharge location and gap width. [71] It is suggested that the concentration of debris particles suspended in the dielectric in the working gap be measured so that the timing of the discharge and thus its efficiency can be better controlled. [28]

Another process, EDM wire cutting machining,[5,94] is a major recent development in die manufacturing industries. Usually, a copper or brass wire, typically of diameter 0.05 to 0.25 mm, acts as the tool electrode and is wound continuously between two spools at rates of up to 3 m/min. The dielectric, which is usually deionized water, is often injected into the machining zone, coaxially with the wire. By this continuous feeding of the wire, a fresh portion of the tool-electrode is

always presented to the workpiece which is usually clamped onto a machine table. Currents used are typically 2 to 3 A. [53]

The wire-EDM cutting technique finds many applications, for instance, in the manufacture of press tools, dies and even electrodes themselves for use in other areas of EDM.

It is well known that there are many advantages in using the EDM die sinker process to produce dies and moulds. However, the design and manufacturing of EDM electrodes are quite critical. Usually, making EDM electrodes is more difficult than making dies. At present, some new techniques are being introduced for the production of dies and moulds, as for example, the Electrodischarge Machine Centre. This centre includes facilities for process planning, electrodes storage, high speed milling, electrode measurement, EDM, workpiece measurement, and tool change/transport. This system illustrates the future direction of unattended and fully automated EDM technology.

In order to produce EDM electrodes quickly and cheaply, several methods have been investigated, such as using compression of graphite powder to make electrodes. This method is very promising, but requires specialized skill, since particles penetrate the surface of the model, and removing them is very difficult. [37] Another method is the electroforming technique. [21,34,56] The advantages of this method are low cost, since the electroforming process is unmanned, and the quality of the electrode is quite good as the copper is very pure and dense. The disadvantages are the long processing time, and deposition is difficult in deep holes and sharp internal corners. Thirdly, the copper electrodes may be directly produced by a metal spraying process. [37] Here the copper electrode can be made quickly, but the wear during EDM processing is unacceptably high, due to the

porosity of sprayed copper.

1-2-3. Polishing Process for Dies and Moulds

In the dies and moulds industry, the polishing process is also very important in order to obtain a good surface. It is estimated that from 35% to 50% of the total die/mould manufacturing time is devoted to surface finishing or polishing. [45] No matter what processes are used, for instance, CNC milling machining; or electrodischarge machining, polishing is required as the final stage.

The following polishing processes are currently used:

I) Electrical Polishing Methods

One procedure is called Electro-Chemical Arc Machining (ECAM). The ECAM process operates by the combined actions of electrochemical dissolution and electrodischarge erosion. [17] ECAM can offer an alternative method of producing the basic shape of some types of dies and moulds. The advantages are the high metal removal rate of the process with the facility to change easily to an electrochemical finishing mode since both processes share the same working fluid.[44]

Another integrated system has been developed which performs electrode machining, EDM sinking, and electrochemical machining(ECM) finishing, all machining with the same electrode on the same machine. The electrode material, for example graphite, is mounted in the spindle of the EDM machine and milled by a high speed spindle with tungsten carbide tooling operating under

NC control. First the cavity is produced by the EDM process; then the dielectric fluid is replaced by an electrolyte ($NaNO_3$ solution) and the power supply is changed at same time; ECM polishing process is then carried out. It is reported that the heat affected layer from the EDM process was completely removed, and a mirror surface obtained.[67]

The electrochemical polishing process is a very rapid technique. The current, DC or pulse power, is utilized across the electrode and the workpiece. The electrolyte (NaCl or $NaNO_3$) is passed through to the gap. 100 A/cm^2 current density is always used, without high electrolyte pressure.[53] Researchers [68] state that with appropriate, machine settings, an EDM surface of 40 μm Rmax can removed in just a few minutes.

II) Mechanical Polishing Methods

Mechanical polishing machines can polish three dimensional sculptured surfaces. Usually, these machines were designed as a special purpose robot. They have been on the market for almost a decade. However, their economic use still requires considerable skill and experience because the fundamentals of mechanical polishing are far from being entirely understood.

Current commercial efforts to automate the die and mould finishing process can be divided into two groups. The first group can be referred to as the "machine tool" group, for example, the Hitachi Die Polishing Machine, which was developed at Hitachi's Production Engineering Research Laboratory. [88] The basic machine is constructed along the lines of a machine tool. The polishing head itself, which is driven by a 3,600 rpm D.C. motor, is free to trans-

late along the vertical axis. The table to which the workpiece is fastened translates along orthogonal (X,Y) axes, and is also free to rotate about these axes. The translation of the table under the polishing head is controlled in one of two ways. This machine has taken moulds with an initial surface roughness of 20 -50 μm Ra to a final surface finish of 0.05 - 1 μm Ra.

The second major approach centres around the use of industrial robots with articulation. While the articulated arm provides an extremely versatile positioning platform on which to mount a grinding or polishing head, it also poses very difficult problems in terms of vibrational stability and positional accuracy. This approach is used by the polishing machines made by Aida Engineering, Ltd in Japan.

The DPR-5 Die-Polisher marketed by Aida Engineering Ltd. is unique in several respects. Certainly the most obvious difference between this machine and the Hitachi Die Polishing Machine is its jointed arm design. This arm is a planar linkage which allows the tool to travel at a constant height above the workpiece. Attached to this is the polishing head, supported on an air cushion, which can translate a maximum of 40 mm in the vertical direction while the tool is in contact with the workpiece. The entire supporting arm can be adjusted over a range of 300 mm on the central pillar, to accommodate work-pieces of different sizes.

All these methods, including milling machining, high speed machining, electrodischarge machining and polishing processes, have advantages and limitations. Some require advanced equipment, skill and control procedures to be successful. Each method also has its own tolerance range, costing and lead time.

In order to reduce the lead time and the cost of tools, the Brite-Euram project, called Manufacture of Tools and Dies Using Spray Forming Techniques (MUST), has been devised to develop the production of tools and dies by the Simultaneous Spray Peening (SSP) process.[13,76]This project attempts to develop the new technology which can replace conventional processes to produce dies and moulds quickly and cheaply.

1-3. MANUFACTURE TOOLS AND DIES USING SPRAY FORMING TECHNIQUES (MUST)

With the development of die and mould manufacturing industries, more and more companies attempt to find new processes, which can reduce manufacturing costs and lead times, and obtain dimensional accuracy and high quality surfaces for dies and moulds. In order to meet this requirement, the MUST project was undertaken to investigate a novel approach for manufacturing tools used in the gravity die casting and plastics injection moulding industries, using a combination of electroforming and metal spraying.

A feasibility study, conducted under the Brite-Euram programme [83] by Sprayforming Developments Ltd. of Swansea describes the use of a novel metal spraying process, known as Simultaneous Spray Peening (SSP), for the production of coining dies. This process involves building up the die by spraying molten tool steel onto a suitable pattern, whilst simultaneously peening the deposit with steel shot to relieve residual stresses caused by the solidification and cooling of droplets of sprayed metal and to reduce porosity. The significance of simultaneous spray peening is twofold:

i). The tensile thermal stresses, which normally lead to distortion of spray formed deposits, can be neutralised by the compressive stresses, which are built up by SSP. By adjusting the kinetic energy of the incident shot, distortion of high temperature deposits can be eliminated, enabling precise replication to be achieved in a similar manner to that already used in industrial production with low temperature metals using conventional spray deposition techniques.

ii). The porosity of the deposit is almost eliminated by the mechanical work which is imparted at high temperature to the deposit continuously as it builds up.

An essential feature of the process is that the spray deposition is carried out with inert gases in a chamber which maintains an oxygen free environment during the spray forming operation to achieve optimum metallurgical properties, and retains the peening shot during SSP. The chamber used for this work is shown in Figure 1.2 and a schematic representation is presented in Figure 1.3. The dimensions are 600 mm x 500 mm x 700 mm. An arc spray gun is mounted on the top plate as driven by a 0.75 kW motor. The peening is capable of delivering 3 mm shot diameter at rate up to 0.75 kg/s and 30 m/s. The work is 150 mm distant from the arc spray gun. The substrata is manipulated during spray deposition to produce deposits consistent in thickness and profile. [77,78]

The SSP process has shown promising results, but the feasibly study showed several problems concerning the accuracy with which a pattern can be reproduced, tool porosity and pattern materials which need to be overcome before the process can be applied industrially. One method which has been suggested for overcoming these problems is to develop a hybrid Electroforming - SSP process in which the working surface of the tool is formed by a thick electroformed shell which is reinforced by having an SSP - sprayed support of tool steel applied to

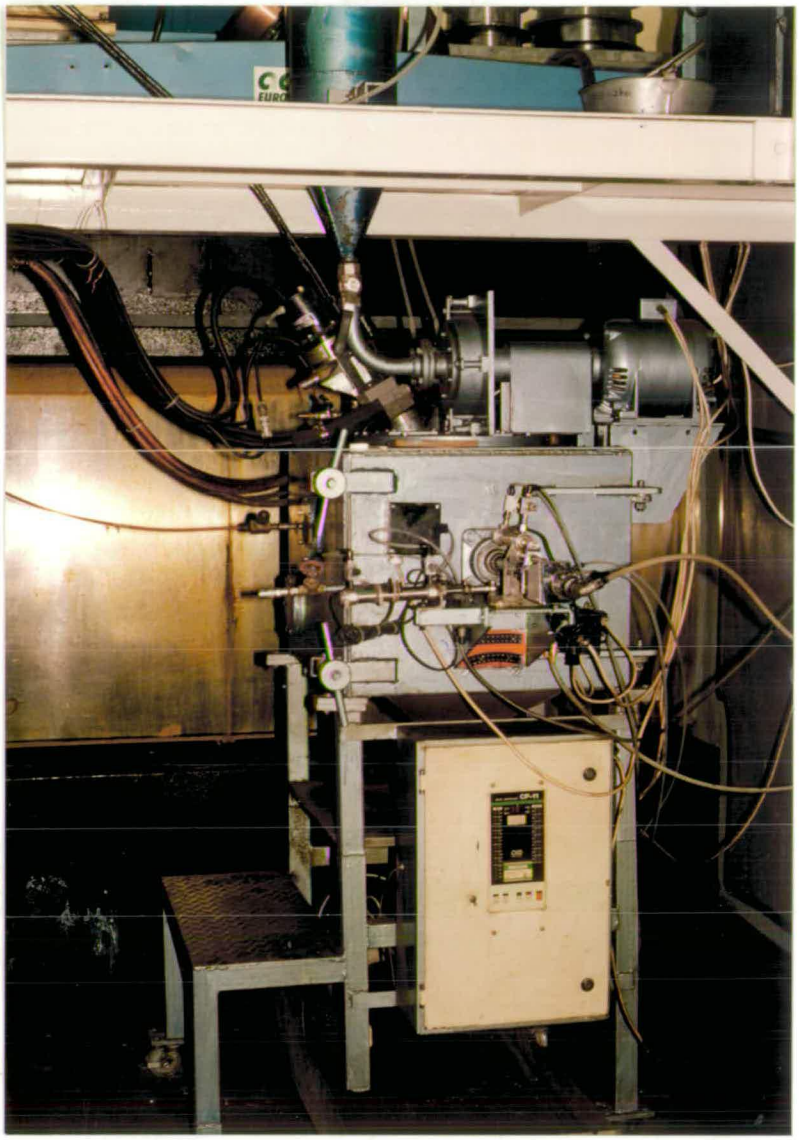


Figure 1.2 View of Equipment Used for Simultaneous Spray Peening

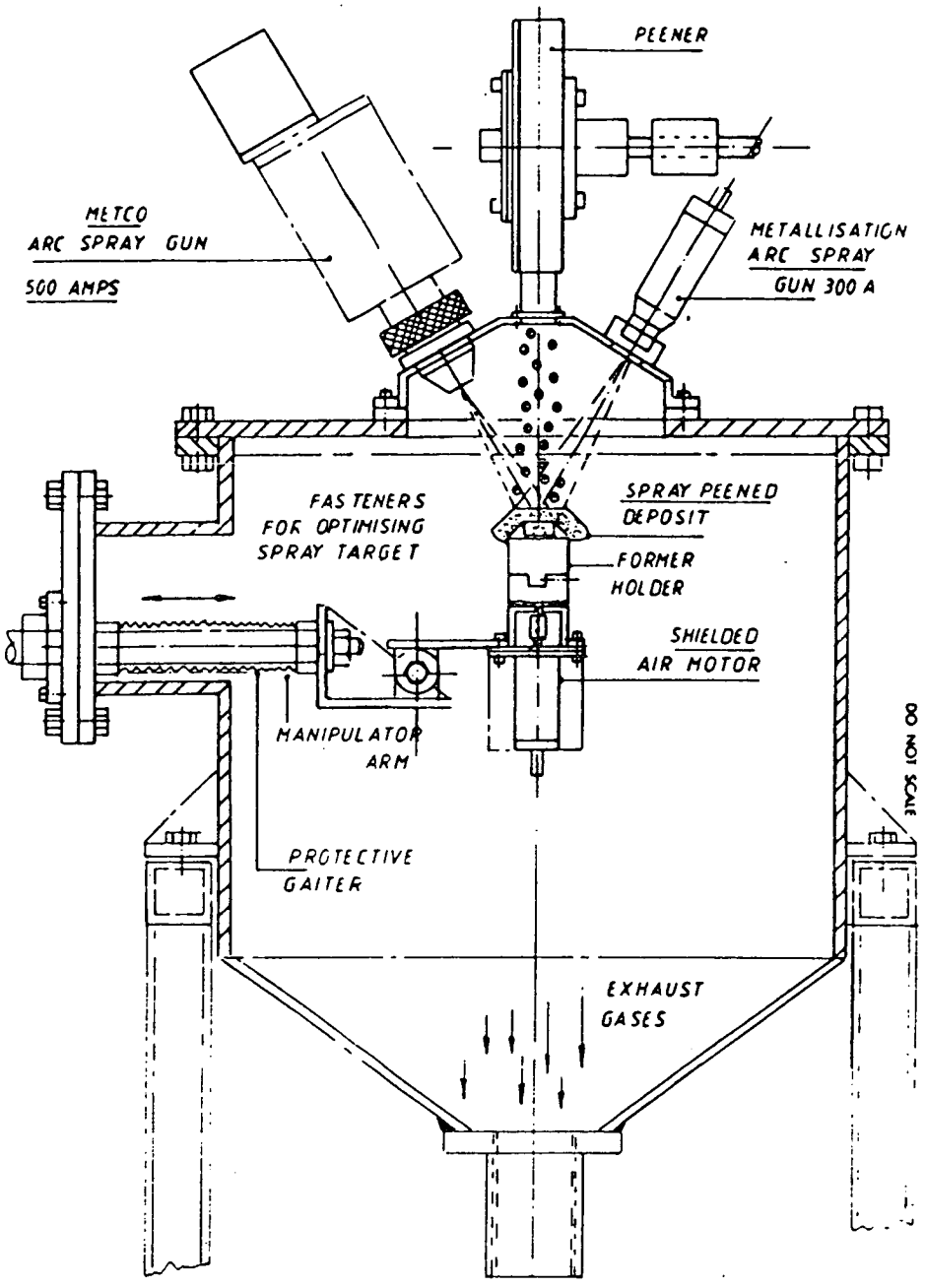


Figure 1.3 Schematic of Simultaneous Spray Peening Equipment

its back (non-working) surface. Although tool steel cannot be used for electroforming it is possible to produce electroformed shells in metals such as nickel and chromium which should have suitable mechanical and chemical properties for use as the working surface of an injection moulding, die casting, or coining die.

The MUST project has been devised further to develop the production of tools and dies by the SSP process. The detailed investigation of the possibilities of the hybrid SSP process will form an important part of this development programme. Research has been undertaken to develop the electroforming aspects of the hybrid SSP process. All techniques and theory of hybrid SSP process will be discussed in this thesis.

It is known that the use of an electroformed shell for the working surface of a composite die has several important advantages over a simple SSP steel die. These advantages are:

1). More accurate reproduction of surface detail and finish.

SSP has been found to be capable of fairly accurately reproducing the surface detail on a small test piece (coin) but with present knowledge it is unable to reproduce surface finish finer than about 1 μm Ra. This is by no means a poor performance, but a properly made electroforming is able to reproduce surface finish down to 0.1 μm Ra, an order of magnitude better than state of the art SSP. This ability to reproduce a very smooth finish is important for plastics injection moulding.

2). Reduced porosity.

The metal sprayed dies produced by SSP have been found to be slightly porous, with densities of 97% to 98% of solid metal compared with 85 - 90%

without peening. [79] This porosity is one factor which limits the ability of the SSP process to reproduce surface finish. It also results in the presence of tiny pits in the surface of the SSP die which can hinder the release of finished components from the die. An electroformed shell, on the other hand, has a density of virtually 100% and consequently is not porous at all.

3). Increased hardness.

The working hardness of 13% Cr tool-steel dies is usually about 630 VHN. This hardness is only achieved after extensive and expensive heat treatment.However, a heat treated electroless nickel shell will achieve a hardness in the range 800-1,000 VHN, and similar values may be obtained using hard chromium or rhodium-faced nickel. The greater hardness of the working face of hybrid dies will result in their having a longer working life than those made of tool steel.

4). Improved corrosion resistance

Nickel is less reactive than steel, and so more resistant to corrosion. Thus a hybrid die will wear less rapidly from this cause than one made from steel alone. This is particularly important for certain plastics moulding applications, such as the injection moulding of PVC, where hot and very reactive chlorine compounds may attack the die surfaces.

5). Wider choice of pattern/mandrel materials

The basic SSP process requires a pattern that is strong enough to withstand peening at an elevated temperature without appreciable distortion or damage.

These requirements effectively limit the simple process to using metal patterns, which may themselves be costly to produce. These patterns are at some risk of damage during the tool making process. However, electroforming operates at lower temperature (from room temperature up to about 90^0C depending upon the metal being deposited). Unlike SSP the process does not impose any mechanical loads upon the pattern. This means that a much wider range of materials may be used as patterns, the only provisos being that they should not react with the electrolyte bath, they must be non-porous, and that they should be capable of being made conductive on their surface so that plating can take place. Alternative pattern materials may include low melting-point alloys, plastics, and silicone rubber. The ability to use a polymeric pattern may be of considerable importance to the manufacture of patterns for the hybrid process by stereolithography.

CHAPTER 2

REVIEW OF ELECTROFORMING

2-1. HISTORY

Electroforming had its beginning in the early 19th century. It was discovered by Professor B.S.Jacobi of the Academy of Sciences, Russia in 4th October, 1838. During his investigations on galvanic cells, Professor Jacobi unwittingly discovered electroforming. Jacobi used an engraved copper printing plate as the cathode in a copper sulphate solution and electrodeposited copper upon it. Although he had difficulty separating the electrodeposit from the engraved printing plate, where it could be separated, the deposit had accurately reproduced the details engraved on the original plate.[31]

In 1842, Professor R.Boettger of Germany successfully electroformed articles of nickel using nickel ammonium sulphate solutions. During the latter half of the 19th century electroforming with iron was investigated. Thus, the three metals, copper, nickel and iron, were electrodeposited and applied to the production of electroformed articles before 1870. [31]

Following its discovery, electroforming was immediately applied to the reproduction of art objects, such as sculptures and statues, and to the duplication of engraved plates for the printing of money because of its capability to reproduce fine surface detail with great fidelity.

Modern applications of electroforming are diverse and today nickel is used in a great variety of different electroforming applications. World consumption of nickel for electroforming is estimated to be 5,000 - 6,000 tonnes per annum. [91] The reasons for its popularity include the fact that electrodeposited nickel can be strong, tough and resistant to corrosion, erosion and wear. Its mechanical properties can be varied by incorporating particles and fibers within the electrodeposited nickel matrix.

2-2. BASIC PRINCIPLE OF ELECTROFORMING

Electroforming is a specialized application of the electroplating process. Its basic principle is similar to electroplating. Electroplating is carried out in a solution with a direct current passed between an anode and the part to be plated which is made cathodic. Usually, the anode is metallic nickel or copper which dissolves under the influence of the current passing, forming nickel or copper ions that replace those discharged in the form of metallic nickel or copper on the cathode. The principle is illustrated in Figure 2.1.[90]

During electroforming, metallic ions and hydrogen always travel to the cathode (mandrel), and negative ions to anode. For example, if two copper electrodes are immersed in copper-sulphate solution, copper is dissolved from the anode, and deposited on the cathode. With carbon electrodes in a brine solution,

hydrogen appears at the cathode and chlorine at the anode. There are two fundamental laws, discovered by Faraday, which are obeyed by all electrolytes.

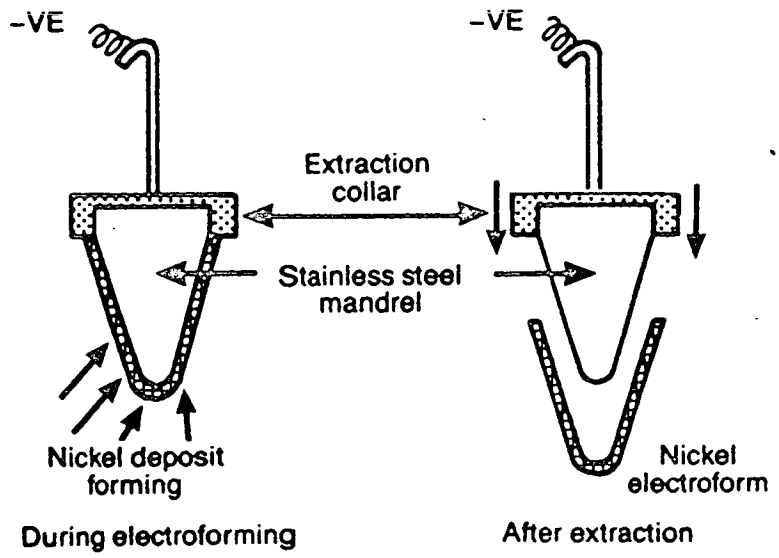


Figure 2.1 Principle of Electroforming [90]

(i). The mass of a given substance liberated at one electrode is proportional to the total charge which has passed.

(ii). The mass of a given substance liberated at an electrode by unit charge is proportional to the chemical equivalent of that substance.[6]

These two laws can be condensed into the following form: If the mass liberated at an electrode is m when a current I passes for a time t, then

$$m = ZIt \qquad (2.1)$$

where Z is a constant for a given element, called the electrochemical equivalent. The chemical equivalent of an ion is the atomic weight divided by the valency, the valency being equal to the number of electronic charges carried by the ion. Then if A is the atomic weight and v the valency:

$$m = (A/v)It/F \qquad (2.2)$$

where F is a universal constant known as the Faraday. The Faraday is the charge of electricity which liberates one gram equivalent (A/v) of an ion in electrolysis.

For example, when an ionic compound dissolves in a solvent, it dissociates into equal numbers of positive and negative ions which are free to diffuse uniformly through the solvent thus:

$$NiSO_4 \text{ ------ } Ni^{2+} + (SO_4)^{2-}$$

However, since they are electrically charged, if we apply an electrical field, they will move under its influence, the positively charged nickel ions going towards the negative electrode (cathode) and the negatively charged sulphate ions going towards the positively charged electrode (anode). Over a range of applied voltage the resulting current consists essentially of two electrons carried by each nickel ion plus two electrons carried by each sulphate ion.

Assume, during electroforming nickel, 2 A current was used for 20 hours. According to formula (2.2), the mass which is deposited on the cathode can be calculated:

$$m = \frac{59}{2} \times \frac{2 \times 20 \times 3600}{96494} = 44\,(g)$$

In practice, a small percentage of the current passing to the cathode causes discharge of hydrogen ions, and hydrogen bubbles are formed. For example, the efficiency of the deposition of nickel is less than 100%. The practical figure is usually 96 to 98%, the exact value depending upon the plating conditions such as pH, current and additives used.[92]

On the other hand, mixing speed also affects the current efficiency. Generally speaking, for a well-stirred solution, the quantity of electricity which can be passed while maximum efficiency is maintained, can be increased by increasing stirring speed. [32]

2-3. ADVANTAGES AND LIMITATIONS OF ELECTROFORMING

It is well known that the electroforming process has been more and more widely applied in industry since 19th century. Why is this process widely used by engineers? Before attempting to answer this question, let us consider the following advantages of electroforming.

(1). The electroforming process can reproduce fine surface detail with great accuracy.[62] For instance, a typical application of electroforming is in the manufacture of microgroove phonograph record, the lateral excursion of the

recording needle cannot exceed about 0.05 mm. This degree of accuracy makes possible the high quality stereophonic recordings available today.

(2). The mechanical and physical properties of an electroforming can be controlled over a wide range by choice of a suitable metal, and by adjusting the plating bath composition and conditions of deposition.

(3). With proper choice of mandrel, parts can be reproduced in quantity with a very high order of dimensional accuracy. The accuracy attainable will depend on the nature of the mandrel material and the accuracy specified, which may be of the order of 0.004 μm. Once the mandrel is prepared, all parts produced from this mandrel will be dimensionally identical.

(4). There is virtually no limit to the size of the object that can be electroformed. Articles have been produced ranging from nickel foil 2.5 μm thick to textile printing screens up to 6 metres long.

(5). Shapes can be made and reproduced that are not possible or very difficult by any other method of fabrication; e.g. seamless radar wave guides with two right angle bends and with the interior being made to close dimensional tolerances and with a high surface finish.

(6). Electroforming is applicable to the making of single pieces, or large mass production runs. In the latter case, it is possible to produce one master, which may be very expensive, and build up successively in several generations a number of negatives and hence positives; these positives are used to make more negatives, thus increasing the production rate until the desired capacity is reached.

(7). In the specific case of moulds and dies for making plastics, zinc and glass parts, electroforming can provide tooling with resistance to corrosion, erosion and abrasion, with good heat conductivity and precise parting lines to minimize and eliminate flashing; and with high wear resistance over long production runs.

While there are many advantages of electroforming processes, we must also point out the typical restrictions of the technique.

One of the main difficulties is associated with the throwing characteristics of an electroplating operation. The deposit produced upon a recessed area of a surface is significantly thinner than on the rest of that surface. Thus it may become impossible to produce a deposit in deep recesses. This problem will be discussed in Chapter 5, section 5-3-2.

Corner weakness is another major defect restricting the successful electroforming of components containing recessed areas. [39] The metal deposited into such sharp angles will have a plane of weakness along the line which bisects the angle. If a radius has been provided, the weakness will extend from the centre of this radius. If the mould has to support a large stress on such lines of weakness, there is a serious danger of early failure during its use.

Comparing with conventional processes, such as lathing, milling and grinding, the electroforming process is very slow, often being measured in days. In addition, the cost of an electroformed article may be relatively high compared with a mass produced component.

2.4. APPLICATIONS OF ELECTROFORMING

Applications of electroforming may be classified as following:

a. Manufacture of duplicating plates:

These applications include electrotypes, phonograph record masters, and security printing plates. Watson [89] reported nickel electroforming was used in the production of printing plates for bank notes. At the United States Bureau of Engraving and printing, more than 40 tonnes of nickel is consumed to make the plates needed each year for production of six billion bank notes and 40 billion postage stamps.

b. Thin - Walled Section:

Foil [54], sheet, hypodermic needles, fine - mesh screen [69] and seamless tubing are also produced by the electroforming technique. For example, electroformed nickel mesh products comprise a large number of current applications. Chief among these are textile printing screens which are used to produce multicolored patterns on textiles, wallpaper and carpeting. The most popular printing screens are seamless electroformed cylinders of nickel consisting of mesh with many fine, precise holes. The designs are created on the screen mesh by photoresist techniques which block some of the openings and leave others free. The screens are mounted on rotary textile printing machines on color feed tubes which are inside and concentric with the large screens. Other mesh products which have been electroformed include filters and sieves, and electric razor screens.

c. Parts difficult or impossible to make otherwise:

These are radar wave guides, linear electron accelerator structures, surface roughness gauges, computing cams, and fountain pen caps etc. In the aerospace industries, electroforming technology is used to manufacture the lightweight precision parts, such as waveguides, antennae, and rocket thrust chambers.[2].

d. Precision parts:

Some tools can be produced by the electroforming process for plastic injection moulding. [25,84] With the introduction of nickel as a material for producing electroformed moulds and the great variety of mechanical properties that can be obtained by controlling the condition of electrodeposition appropriately, there is practically no restriction on the use of electroformed material for moulding operations. Electroformed moulds have been used successfully for moulding thermosetting compounds, for injection moulding of thermoplastics, for the die casting of zinc or low fusible alloys, for the pressing of ceramic material and even for the pressing of glass. [4]

There are several methods to support the electroformed shell. Some researchers [19,52] reported that they are successfully using epoxy resin; cast iron; cast lead alloy and sprayed steel. Each has advantages and disadvantages.

2.5. EXPERIMENTAL APPARATUS

Electroforming is a technique which is used to produce actual components employing the same basic equipment and practices as in conventional decorative plating.

The simplest method involves a mandrel or former which is coated with a substantial layer of metal and when the mandrel is removed, the electrodeposit is left as a self - supporting component in its own right.

The electroforming equipment consists of a tank; the plating solution; stirrer; anode (often of same composition as the metal being deposited); the mandrel as cathode; a heater with thermostatic control; and a power supply with ammeter, voltmeter and stepless control. In order to meet the present experimental requirements, a PVC tank was supplied by Engineering Services for Plating Ltd. The tank's size is 280 x 280 x450 mm (LxBxD) with the appropriate pump, filter and heater, which can control the temperature of the solution to $\pm 2\ ^{0}C$. The basic schematic is shown in Figure 2.2. The D.C power supply was also provided by E.S.P Ltd. This power supply can provide up to 100 A current and 15 volts with stepless manual control.

Since this tank has a capacity of 30 litre, frequent changing of the electroforming solution would be wasteful. However, a three litre beaker is often used instead of the PVC tank. In this case, a hotplate with magnetic stirrer is applied to maintain the operating temperature and agitate the solution.

In order to improve the electroforming shell quality, a special pulse power supply was designed. This pulse supply can provide square wave output from 20 Hz to 20 kHz without the need to switch range. The Duty Cycle (see

chapter 4) can also be adjusted separately from 30% to 70%. This pulse power supply will be discussed in Chapter 4.

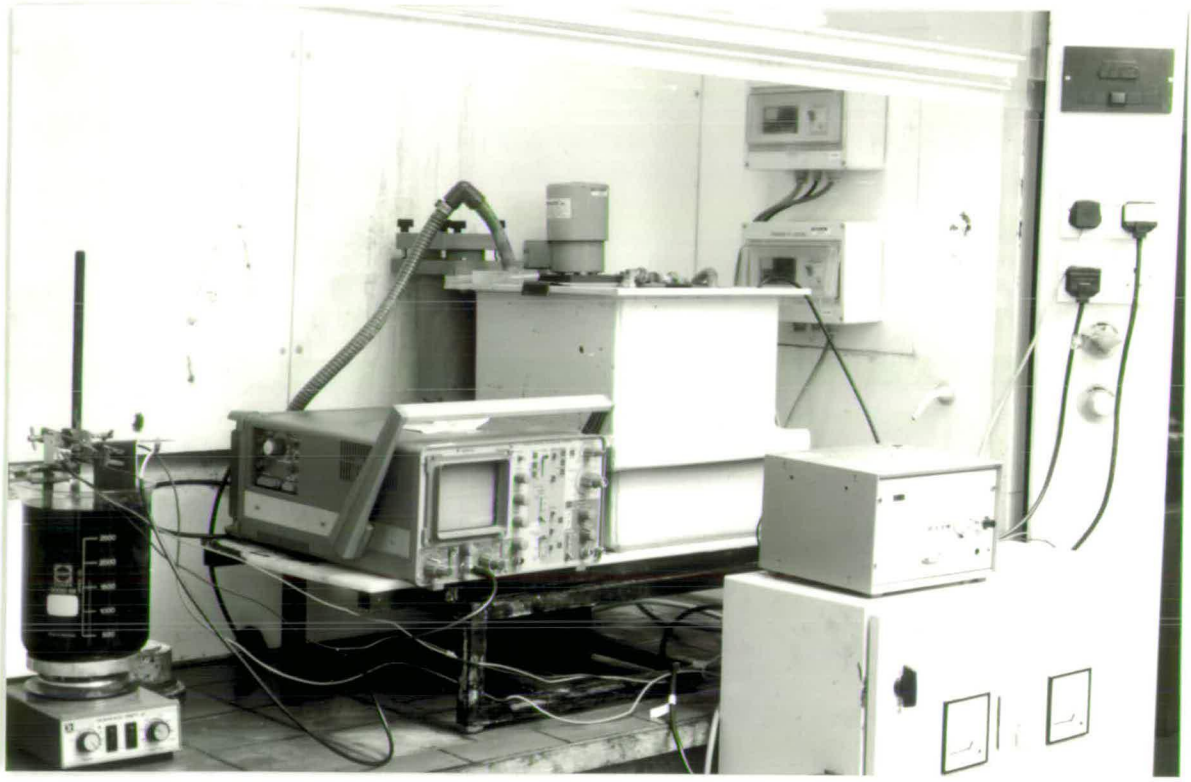


Figure 2.2 Equipment Used for Electroforming

PART TWO - EXPERIMENTAL PROCEDURES AND RESULTS

CHAPTER 3

MECHANICAL PROPERTIES OF ELECTROFORMED NICKEL ALLOYS

3-1. INTRODUCTION

The electroforming industry has for years utilized metals for various purposes without fully realizing the extent to which each application of the metal is based upon its properties. The main properties which determine the value of a thin metallic coating, usually applied for decorative purposes or corrosion resistance, are surface appearance including colour and reflectivity, hardness and abrasion resistance. However, the properties which are of value in heavy electrodeposits, for engineering applications, such as electroforming, are somewhat different. Here the important properties are mechanical, such as internal stress, tensile strength, hardness and ductility. It is well known that the properties of metals can be varied considerably by metallurgical treatments; the properties of electrodeposited metals also can be varied by changing the conditions of deposition. Some of these variations are quite striking, as illustrated in the

following study of nickel deposits. For example, the deposit from a hard nickel solution can achieve the very high hardness of 600 HV(200g).

The main objective of this study was to accumulate data on these mechanical properties of nickel electrodeposits which can be used to produce electroformed moulds. The study involved measurements or determinations of the following: internal stress, hardness, tensile strength and elongation. The effects of heat treatment on many these properties were investigated, and electroless nickel deposits were also examined.

3-2. MEASUREMENT OF HARDNESS, TENSILE STRENGTH AND ELONGATION

The mechanical properties of the deposit are most important for electroformed parts and must be selected and controlled to suit their operational requirements. Many researchers have studied the effect of conditions of deposition and bath composition on the hardness of nickel deposits [8,29,31,85] but no published investigation of mechanical properties of thick electroformed material has been found. In order to prepare samples suitable for the measurement of mechanical properties of the thick nickel, a special small PVC jig was designed and is shown in Figure 3.1.

An aluminium sheet cathode was fitted between two PVC sheets, the front one being machined by milling to expose a tensile test specimen shape. After ten hours electroforming, the test specimen with this shape, which is shown in Figure 3.2, was produced, the thickness being about 1 mm. The specimen is quite easy to separate from the aluminum sheet. The cross-sectional areas of the specimens

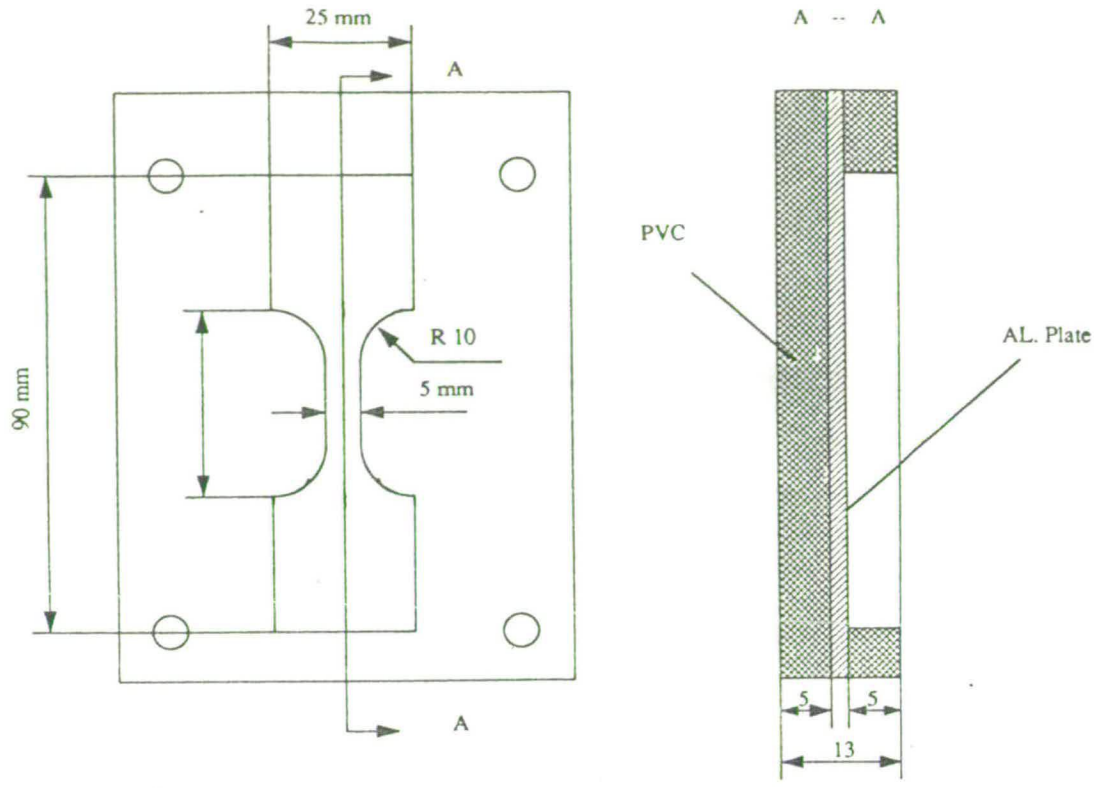


Figure 3.1 Schematic Drawing of Small PVC Jig

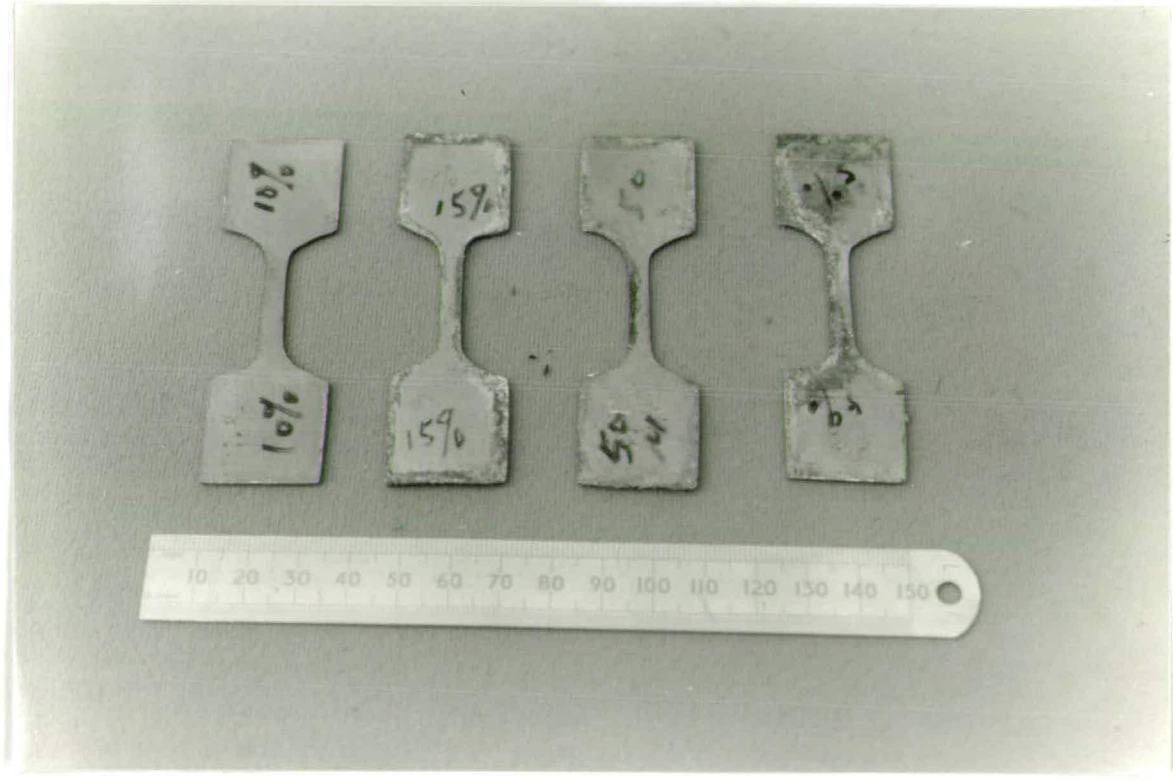


Figure 3.2 Specimens for Tensile Stress Test

were calculated from measurement of their dimensions. The specimens were produced in several differing solutions, whose compositions and operating conditions are shown in Table 3.1. The specimens were tested for tensile strength in a JJ Lloyd M30 instrument. The test instrument and clamping system are shown in Figure 3.3 and Figure 3.4. The maximum loads and extensions were recorded. Then according to the following formulae,[11] the tensile stress and elongation can be calculated:

$$\sigma = \frac{F}{A} \tag{3-1}$$

where σ is the tensile stress; F is the maximum load applied perpendicular to the specimen cross section; and A is the original cross-sectional area before any load is applied.

$$\varepsilon = \frac{l - l_0}{l_0} \quad 100\% \tag{3-2}$$

where ε is elongation, l_0 is the original length before any load is applied, and l is the final length (elongation is usually expressed as a percentage).

The hardness tests at room temperature were made with a Vickers Microhardness instrument (Leitz Miniload 2), with a Vickers diamond indenter and a load of 200 g. The hardness values reported represent an average of five measurements. The hardness was measured again after being heat treated for 1 hour at temperatures up to 500 ^{0}C.

Figure 3.3 JJ Lloyd M30 Tensile Test Instrument

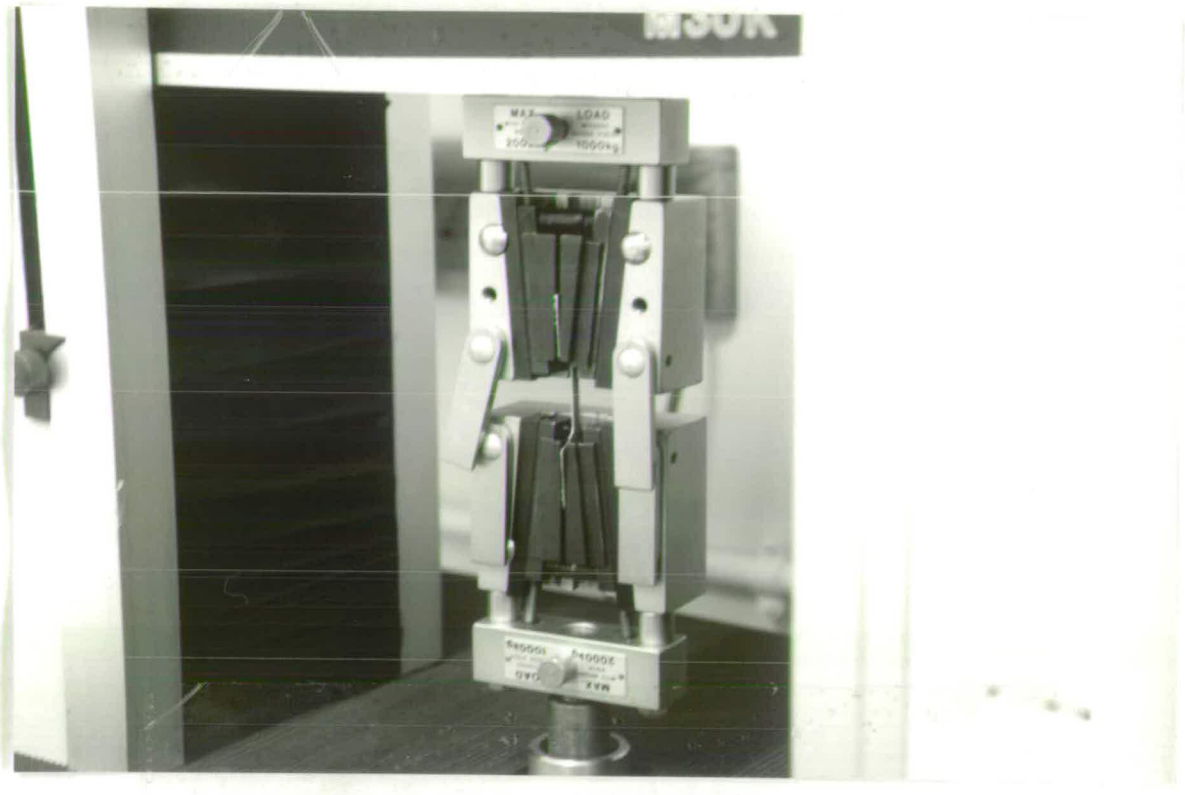


Figure 3-4 Clamping System for Tensile Stress Test

No.	Bath Type	Nickel Sulphamate $H_2(NSO_3)_2Ni.4H_2O$ (g/l)	Nickel Chloride $NiCl_2.6H_2O$ (g/l)	Boric Acid $H3BO_3$ (g/l)	Cobalt Sulphate $CoSo_4.xH_2O$ (g/l)	EPC 30 Agent (ml/l)	Nichem X (ml/l)	Nichem XY - Y (ml/l)	Current Density (A/dm^2)	pH	Temperature (^0C)
1 2 3 4 5 6 7	Nicke Sulphamate	300	0 10 20 40 60 80 100	40					5	4.0	50
8 9 10 11 12 13	Nickel Cobalt	300	20	40	1 3 5 10 15 20				5	4.0	50
14 15 16 17 18 19	Hard Nickel	300	20	40		0.5 1 2 3 4 5			5	4.0	50
20	Electroless Nickel						90	250		4.7	90

Table 3.1 Composition of Nickel Plating Solutions and Operating Conditions

3-3. RELATION BETWEEN MECHANICAL PROPERTIES AND CONDITIONS OF DEPOSITION

It is well known that the mechanical properties of electroformed nickel can be influenced by the composition of the bath and its operating conditions: pH value, temperature and current density. As with conventional metals, the properties are interrelated and steps taken to increase the hardness of a deposit usually increase its strength and lower its ductility.[8,31,43,46]. The refinement of crystal structure, for example by the use of an addition agent, is accompanied by increased hardness and tensile strength, and reduced ductility. Some researchers have reported the effect of bath composition on mechanical properties.[30,56,61,62] George[31] summarized the influence of operating variables on some of the properties of nickel deposited from Watts and conventional nickel sulphamate solutions, which is shown qualitatively in Figure 3.5. [31]

In order to produce high hardness and tensile strength nickel dies, the nickel cobalt and hard nickel solution were investigated. The relationship between mechanical properties will be discussed.

3-3-1. Effect of Bath Composition

The mechanical properties of the nickel deposits are shown in Figure 3.6 and Figure 3.7, which contrast the hardness, tensile strength and elongation of the deposit from nickel cobalt and hard nickel solutions with change in composition. It is very clear that the peak occurs at 5 g/l cobalt sulphamate in the deposit and the hardness is about 357.2 HV (200g), the ultimate strength is about 900±20MPa and 8.7% elongation. With increasing cobalt sulphate, the hardness decreases to

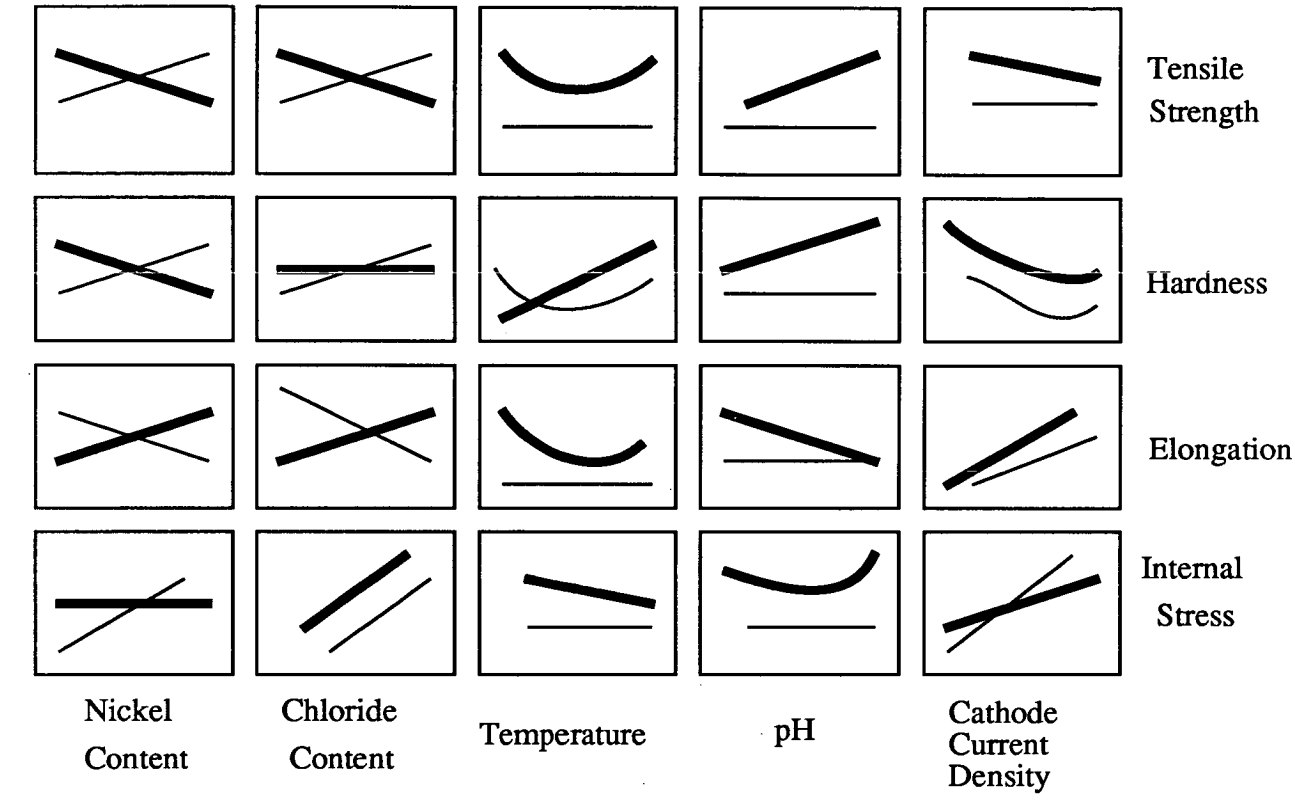
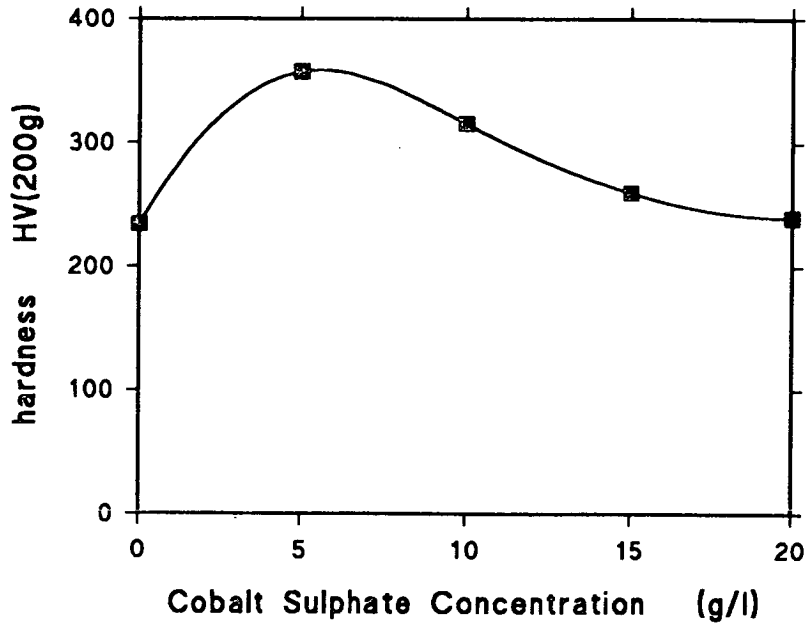
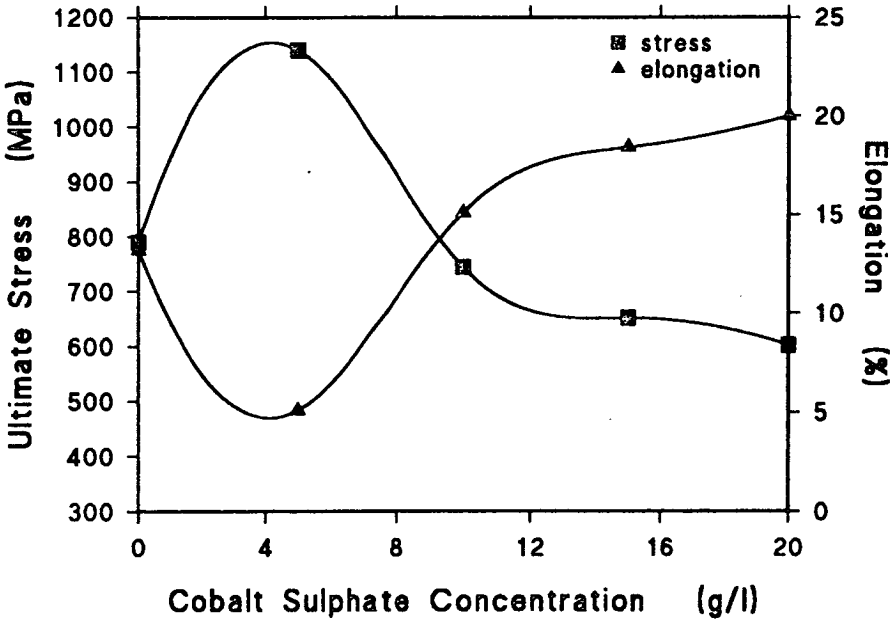


Figure 3.5 Qualitative Effects of Operating Conditions on the Properties of Nickel Electrodeposits from Watts and Sulphamate Solutions [31]

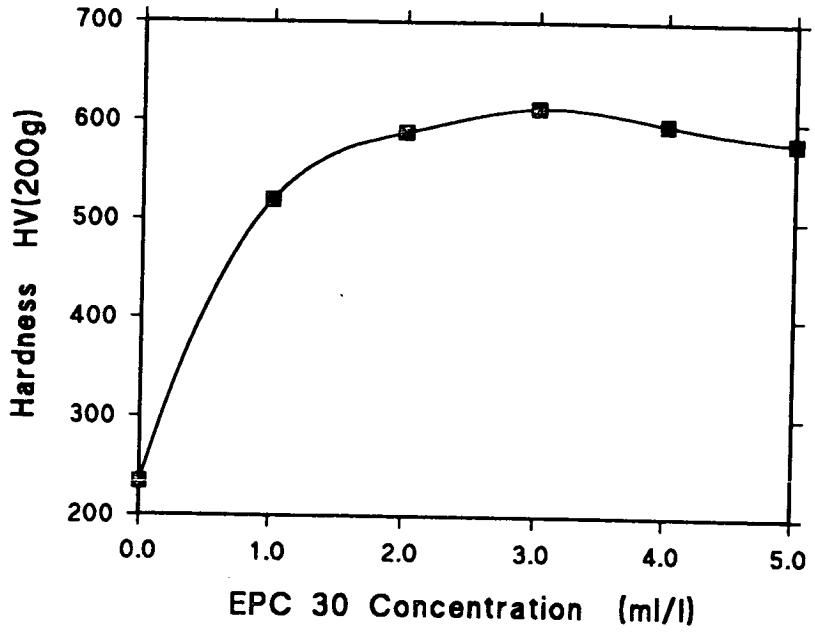


(a)

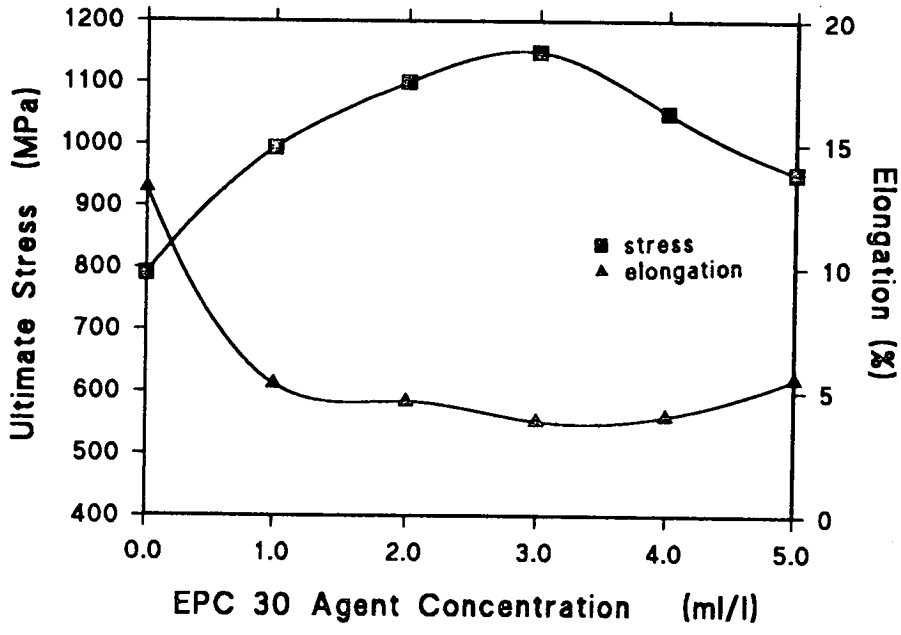


(b)

Figure 3.6 The relation of Cobalt Sulphate Content to Mechanical Properties (a) for hardness (b) for ultimate stress and elongation at 5 A/dm^2, pH 4 and 50 ^{0}C temperature at Nickel Cobalt Bath



(a)



(b)

Figure 3.7 The Relation of EPC 30 Agent Concentration in Hard Nickel Bath to Mechanical Properties (a) for hardness, (b) for ultimate stress and elongation at 5 A/dm^2, pH 4 and 50 ^{0}C

about 240 HV (200 g) the ultimate strength down to 515 ±10MPa and elongation up to about 12%. The great influence of EPC 30 agent on the mechanical properties is represented in Figure 3.7. With over 3 ml/l, the hardness and ultimate stress values decline. With the optimum addition, 3 ml/l EPC 30, the hardness reaches 613 HV, the ultimate strength increases to 1,050 ±20MPa.

3-3-2. Effect of Operating Conditions

The bath composition is the most important factor controlling the mechanical properties of nickel deposits but the optimum operating conditions, though secondary, must be selected if the best results are to be achieved. Figure 3.8 and Figure 3.9 show the relationship between cobalt sulphate concentration, EPC 30 content, and deposit hardness, as a function of current density and solution temperature. In all cases, the effect of increasing current density and temperature is to increase the deposit hardness, though the effect is sometimes small.

It is commonly believed that an increase in current density increases the hardness of nickel deposits and lowers ductility.[7] Our data conform with this fact. The curves in Figure 3.8 show the small variation which occurs with the increase of current density from 5% to 20% cobalt sulphate. Also, the curves in Figure 3.9 show very little change with increase of bath temperature.

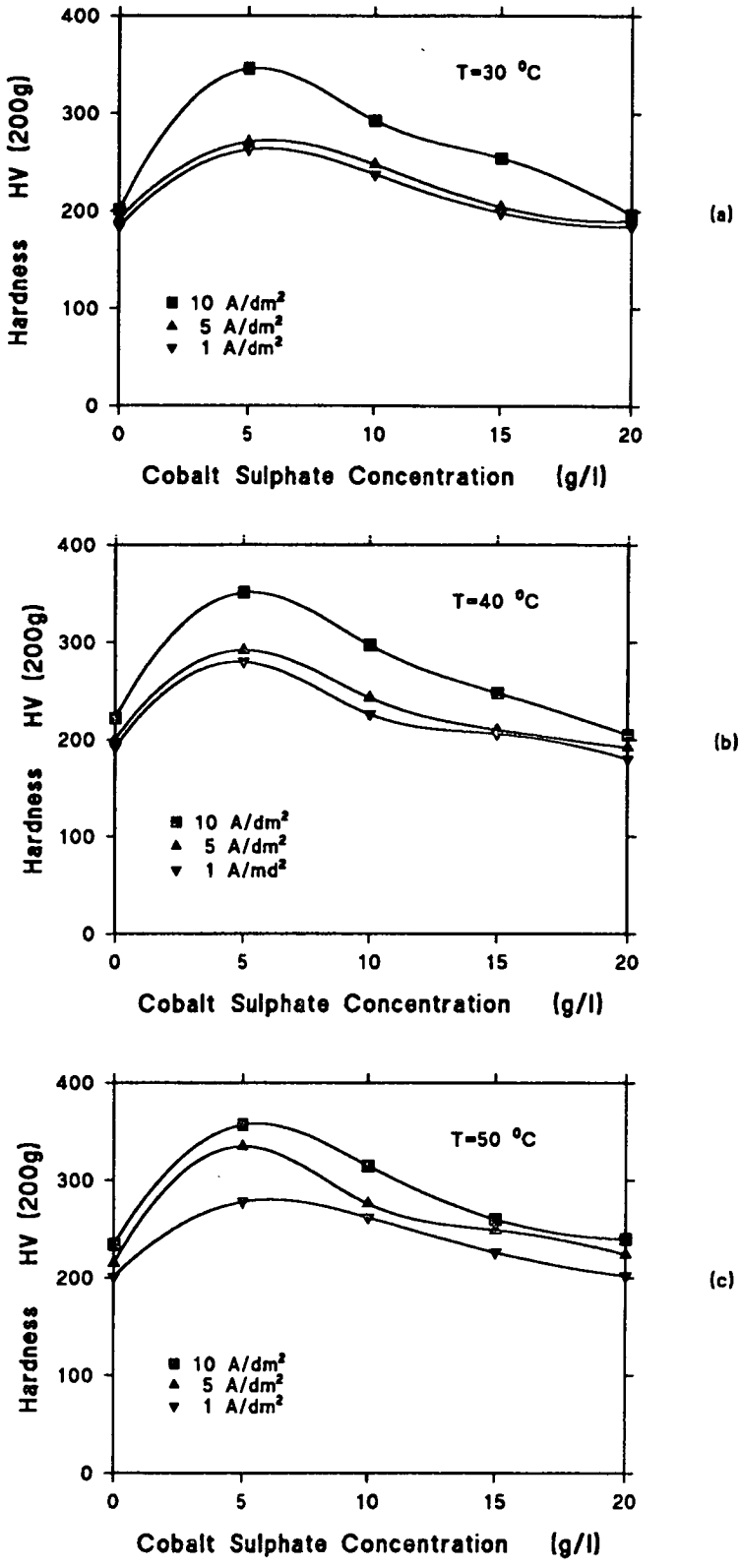
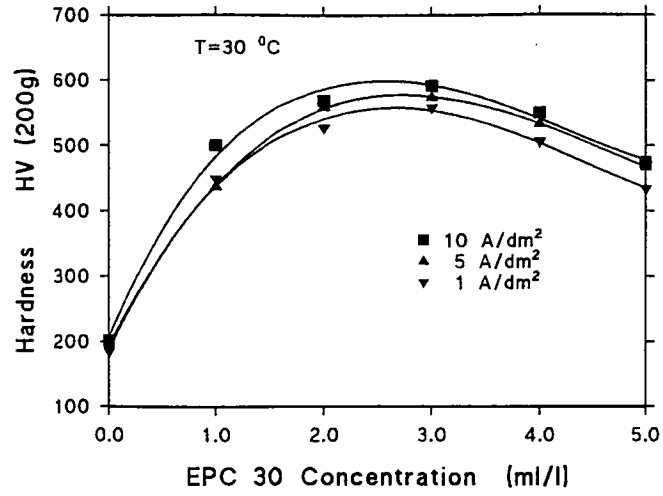
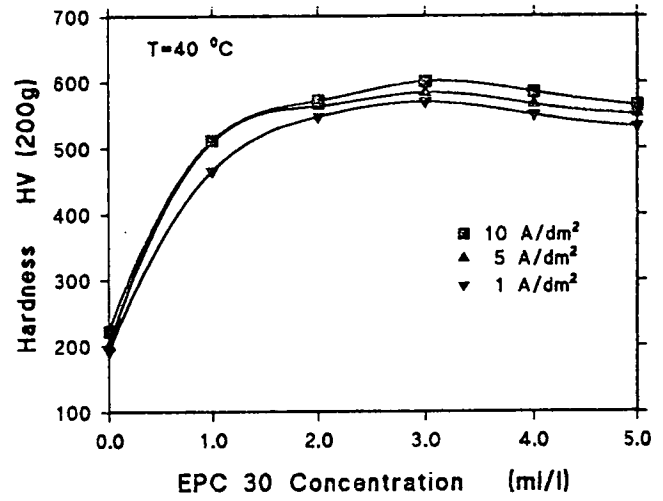


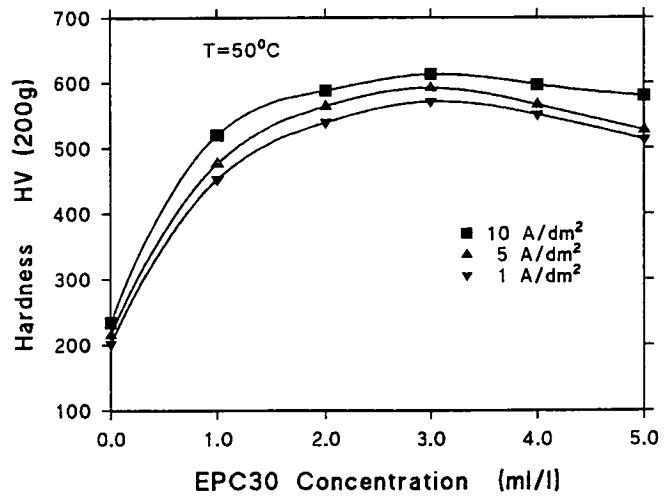
Figure 3.8 Relationship Between Cobalt Sulphate Content and Hardness as a Function of Current Density and Temperature of Nickel Cobalt Bath



(a)



(b)



(c)

Figure 3.9 Relationship Between EPC 30 Content and Hardness as a Function of Current Density and Temperature of Hard Nickel Bath

3-3-3. Effect of Heat Treatment

The mechanical properties, and to a lesser extent the physical properties of metals may vary with the heat-treatment. Electrodeposited nickel may be affected to a greater extent than metallurgically prepared metals, because in the as-deposited condition they may have greater internal stress, smaller grain size, and foreign material in a finer state of dispersion. A simple heat-treatment, which involves only heating to a given temperature followed by slow cooling, relieves stresses within the metal and may cause grain growth as well as agglomeration of foreign material.[9] These changes in the nature of these mechanical properties are dependent upon or related to the crystal structure of the deposit.

The specimens were produced using same process which has been described in section 3-2. Before measurement, the specimens were heat-treated for one hour in a furnace at temperature from 100 to 500 ^{0}C. Then, the mechanical properties, - hardness, tensile stress and elongation -, were separately measured. The results are shown in Figure 3.10 to Figure 3.15.

The results show that increase in heat-treatment temperature over the range 200 to 500 ^{0}C results in a decrease in tensile strength. In fact, for temperatures greater than 300 ^{0}C, the tensile strengths, of the nickel cobalt samples are fairly close. For example, at 400 ^{0}C the value is 540 ±10MPa for 5% cobalt sulphate, at 500 ^{0}C the value is 516 ±10MPa.

Room temperature elongation, on the other hand, increases with annealing temperature, reaching a maximum of 24% at 500 ^{0}C for nickel cobalt and 18% for hard nickel. The effect of temperature over the range 200 to 500 ^{0}C causes a decrease in hardness. The decrease is small for hard nickel up to 200 ^{0}C, but

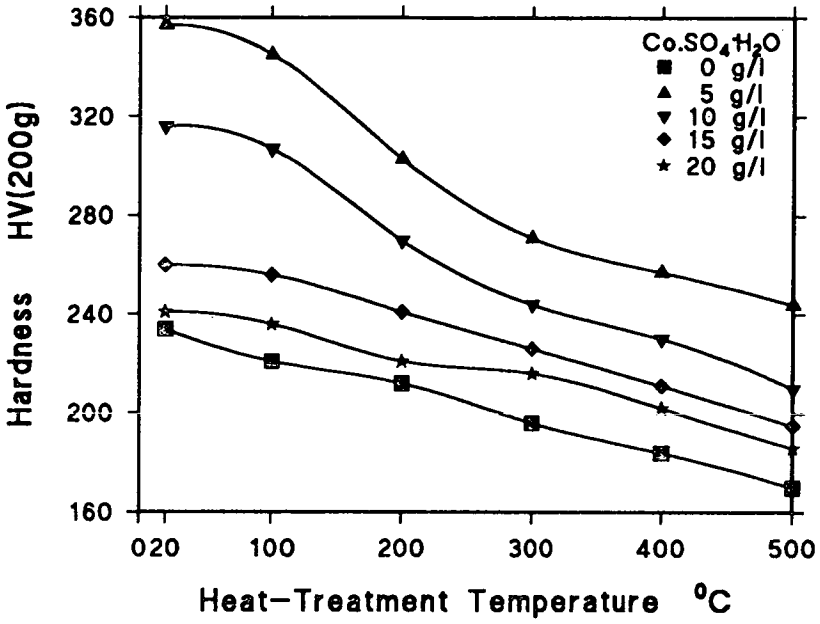


Figure 3.10 Relationship Between the Hardness and Heat-Treatment Temperature for Nickel Cobalt Bath

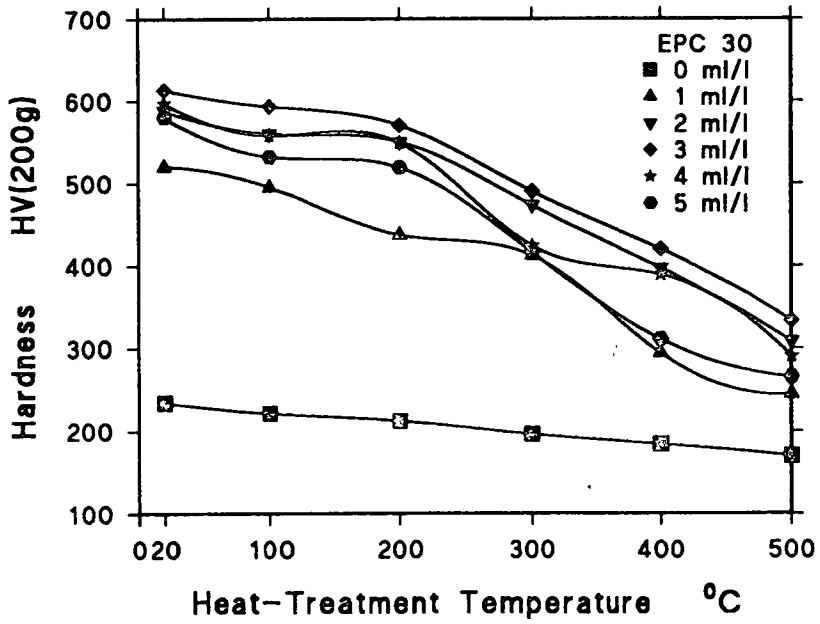


Figure 3.11 Relationship Between the Hardness and Heat-Treatment Temperature with EPC 30 Bath

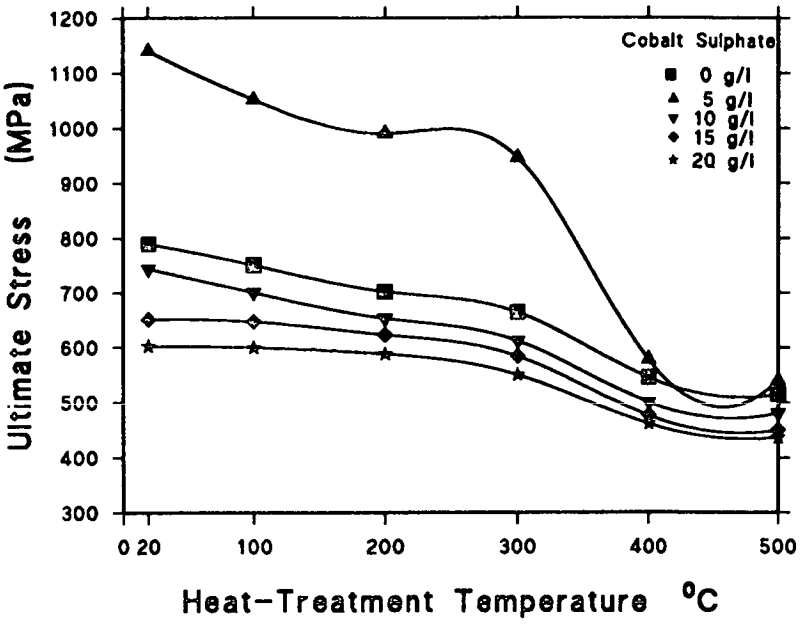


Figure 3.12 Relationship Between Ultimate Stress and Heat-Treatment Temperature for Nickel Cobalt Bath

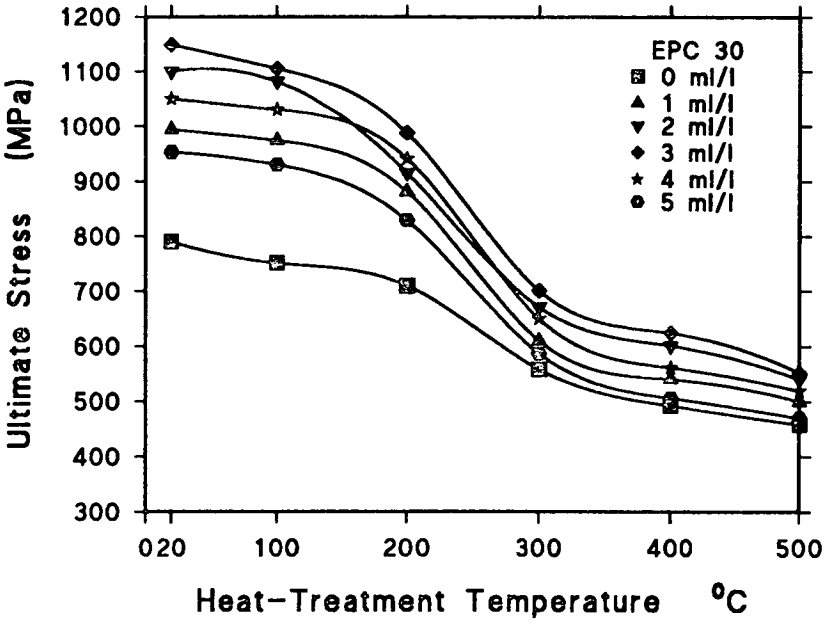


Figure 3.13 Relationship Between Ultimate Stress and Heat-Treatment Temperature for Hard Nickel Bath

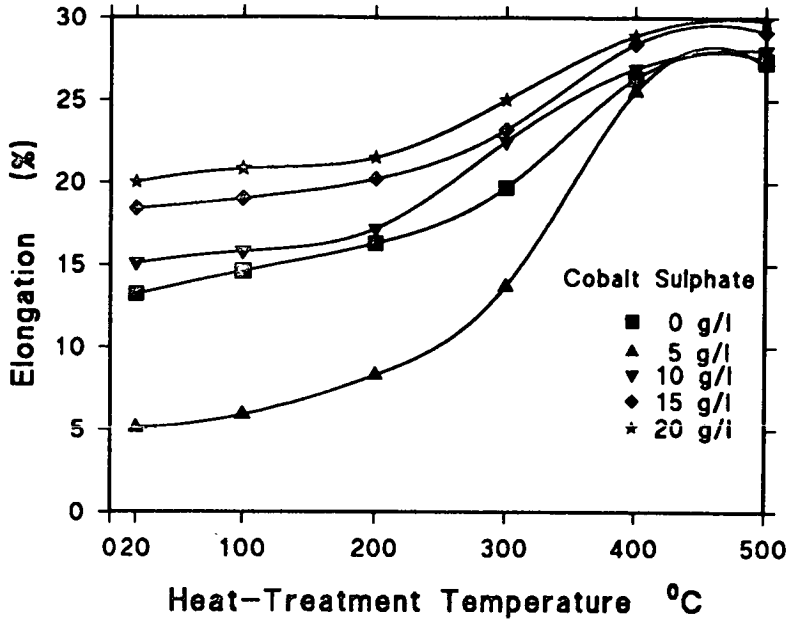


Figure 3.14 Relationship Between Elongation and Heat-Treatment Temperature for Nickel Cobalt Bath

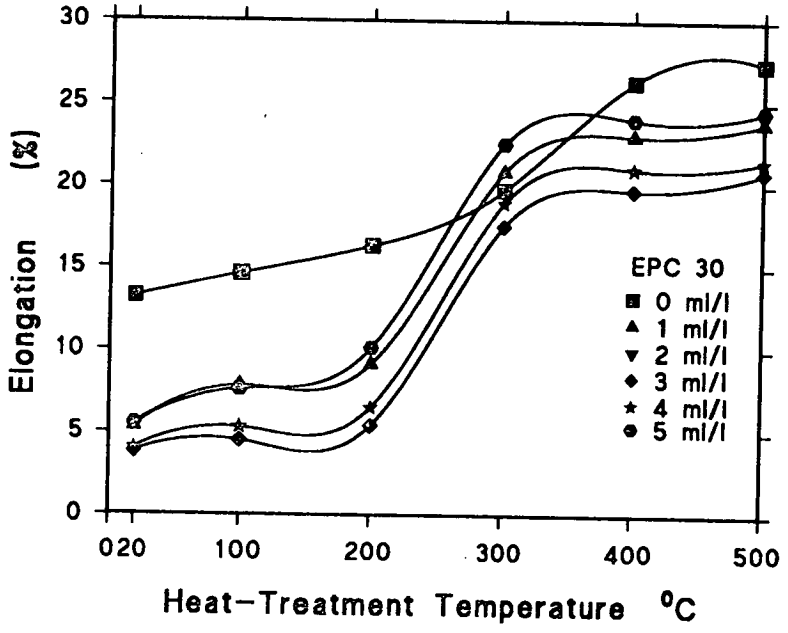


Figure 3.15 Relationship Between Elongation and Heat-Treatment Temperature for Hard Nickel Bath

is greatest between 200 and 500 ^{0}C. The hardness at 5% cobalt sulphate and 3ml/l EPC 30 is higher than other. For instance, at 500 ^{0}C, the values are 226 HV (200g) for 5% cobalt sulphate, and 332 HV for 3ml/l EPC 30 agent.

The change in mechanical properties brought about by heat treatment has been studied by a number of investigators,[47,85,92] who were in general agreement that these changes are related to the changes in crystal structure. All nickel deposits when heat treated undergo a change of crystal structure, the extent of which depends upon the temperature.

In this section, the deposited nickel specimens were heat treated at different temperatures from 100 ^{0}C to1,000 ^{0}C. The period of heat-treatment was one hour at the specified temperature. Then, specimens were section by electrodischarge machining. After ultrasonic washing, mounting and polishing of the specimens, crystal structure analyses were undertaken on the scanning electron microscope (Cambridge Stereoscope 250-S). The results are shown in the following photomicrographs (Figure 3.16 and Figure 3.17).

It can been from Figure 3.16 and Figure 3.17 that the deposit showed initally the characteristic fine grained, laminated structure. This structure could be seen even after a heat-treatment at 400 ^{0}C, but a slight tendency for grain growth was also observable; at 600 ^{0}C part of the fine structure still persisted, but the tendency for recrystallization into larger grains. With the increase of temperature, the size of grains become larger. At 800 ^{0}C and 1,000 ^{0}C, the deposit had recrystallized fully into larger grains and many pores or inclusions were clearly visible. The two kinds of nickel deposits went through a similar series of changes.

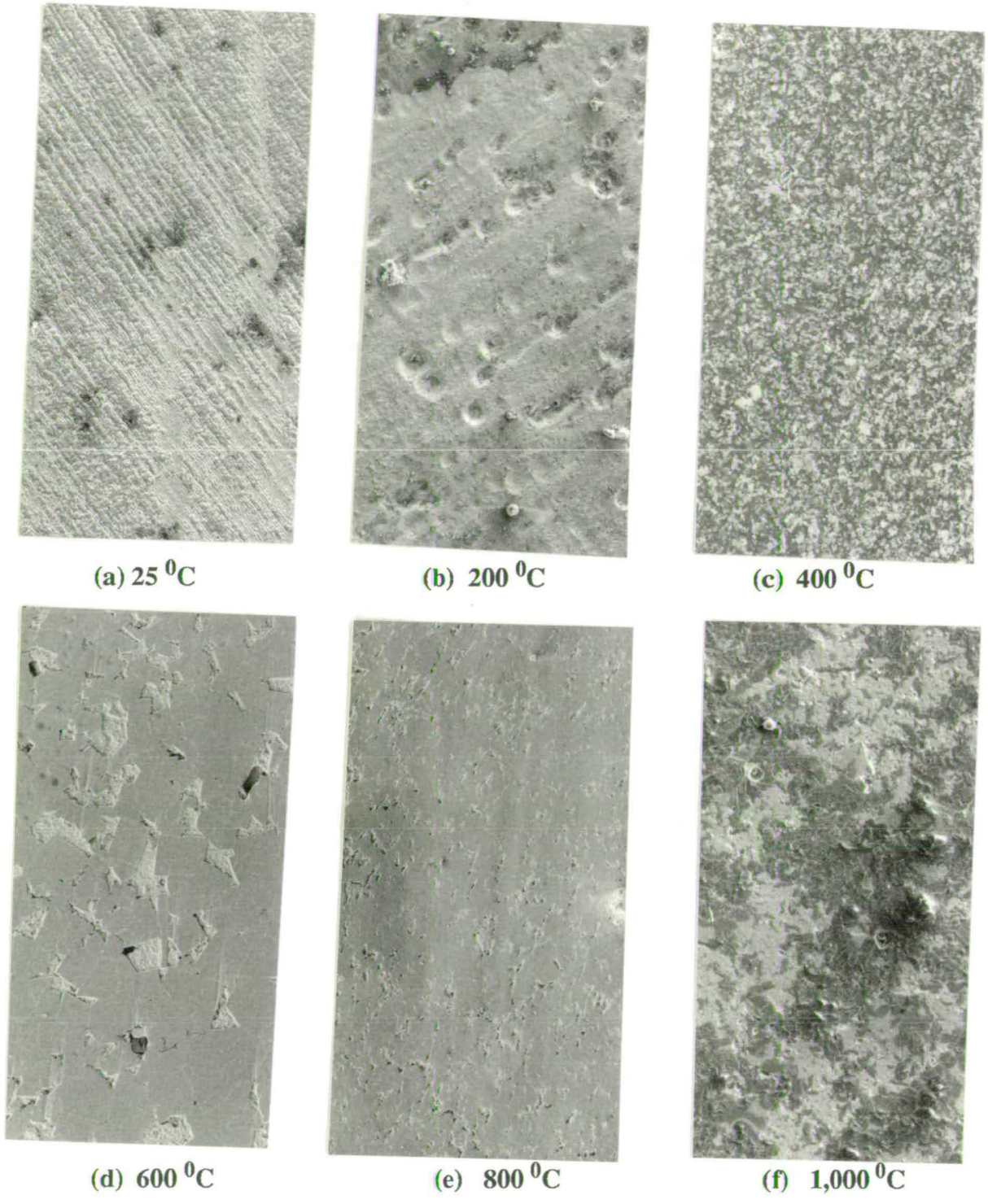
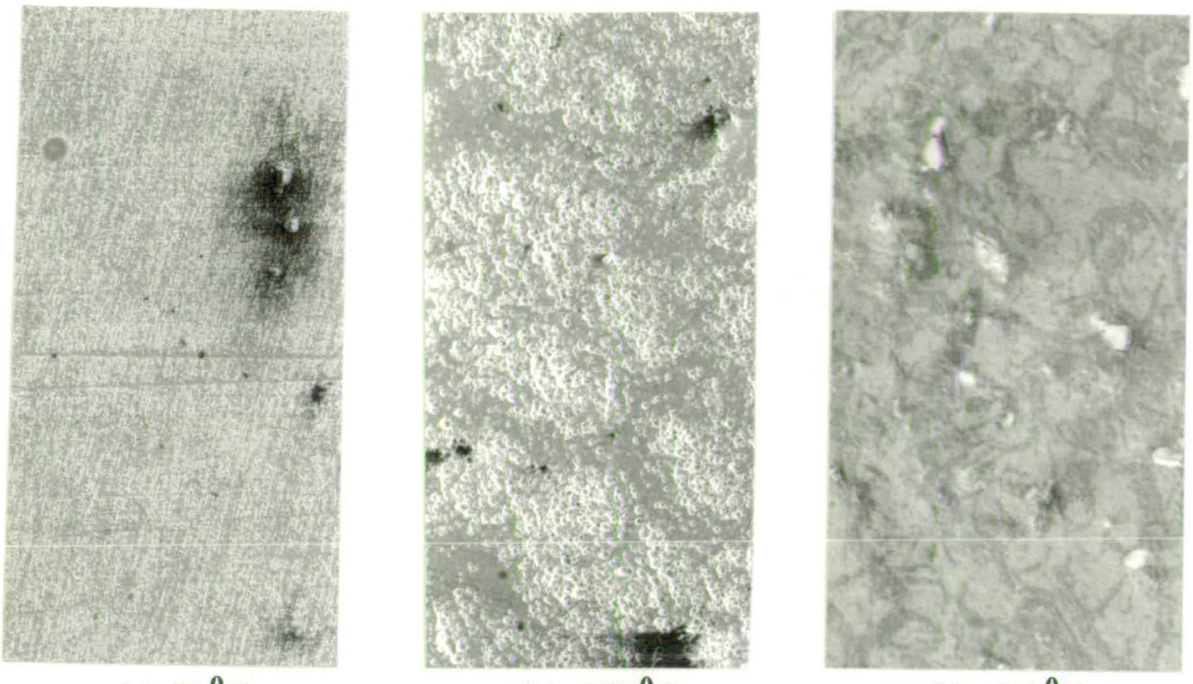
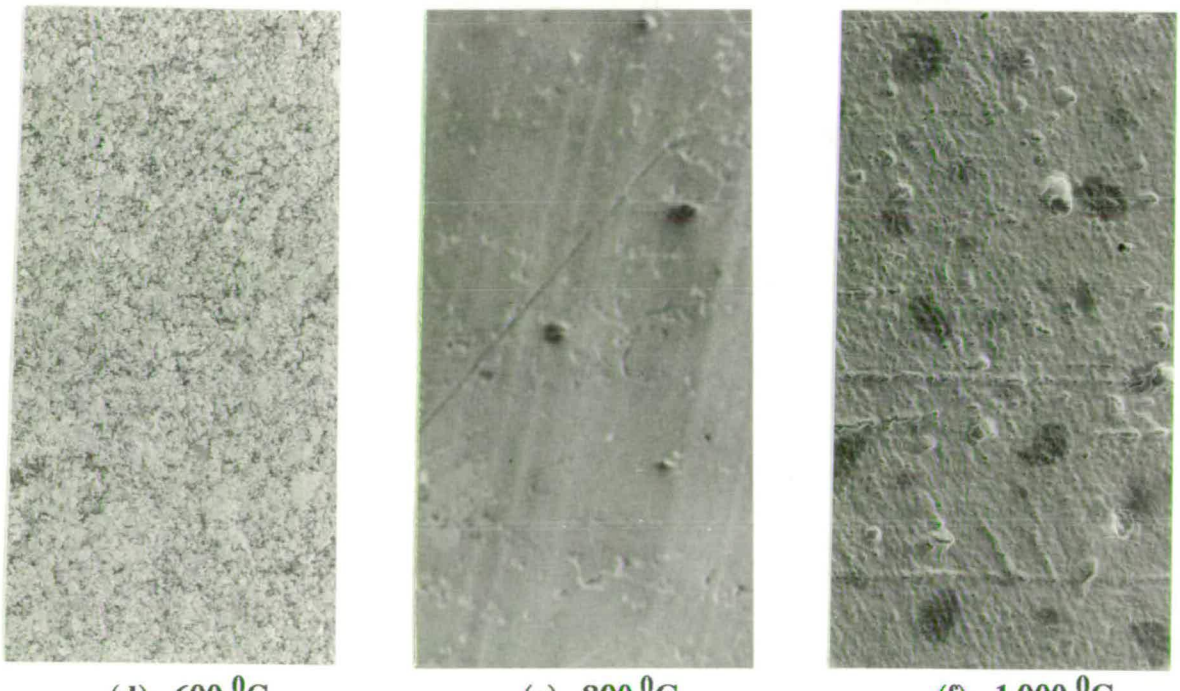


Figure 3.16 Effect of Heat-Treatment on Structure of Nickel Deposits from No.10 Bath at 5 A/dm^2, pH of 4.0 and 50 ^{0}C Temperature, Cross Section x 250



(a) 25 ^{0}C (b) 200 ^{0}C (c) 400 ^{0}C



(d) 600 ^{0}C (e) 800 ^{0}C (f) 1,000 ^{0}C

Figure 3.17 Effect of Heat-Treatment on Structure of Nickel Deposits from No.17 Bath at 5 A/dm^2, pH of 4.0 and 50 ^{0}C Temperature, Cross Section x 250

From these results, it has been recognized that the mechanical properties of electrodeposited nickel were more affected by heat treatment than were other factors, probably because the mechanical properties are more sensitive to changes in crystal structure. This is a main reason that the dies and moulds produced by electroforming could not be used as alloys of higher casting temperature. If the temperature is high enough to change the mechanical properties of nickel layer, such as hardness and tensile strength, the dies and moulds will be damaged. The testing result in section 6-6 had verified this fact.

3-4. INTERNAL STRESS OF NICKEL DEPOSITS

The existence of internal stresses in electrodeposited nickel has been known for many years. "Internal Stress" is an inherent force in an electrodeposit, free from external forces, the deposit being either compressed or stretched as compared with its normal condition. In the tensile stressed condition the deposit has a tendency to contract; in the compressively stressed condition, the deposit has a tendency to expand. [48,95,96]

The control of internal stress is extremely important in electroforming. It is well known that many failures of electroformed parts have been attributed to the presence of highly compressive or tensile stresses in the deposit. High internal stresses can cause peeling or cracking of nickel deposits and distortion of items formed from thin sheet metal. In addition, the dimensional accuracy of an electroformed article and its performance in use may be affected by internal stress. In this study, internal stress was a practical consideration because stressed deposits tend to crack and pull away from the mandrel or warp so badly that the current distribution is not uniform.

Internal stress is influenced by many factors, such as temperature of deposition, current density, pH value, nickel chloride content, nickel content, and addition agent. In this section, the effects of current density, solution composition and temperature will be investigated.

3-4-1. Experimental procedure.

In the presence of a internal tensile stress in the coating, there is a tendency to contraction and if a separation of the coating from the substrate could be imagined, it would have decreased its length. During the elastic interaction between the substrate and the coating, a pair of equal but opposite forces, F, which are trying to equalize the lengths of substrate and coating, are applied at different points, so that a bending moment M is initiated. A schematic diagram is shown in Figure 3.18,[48] (a) for tensile internal stress, (b) for compressive internal stress.

According to the above description, the bent strip method is applied to measure internal stress. Brass strip (40 mm length, 20 mm width and 0.2 mm thickness), which was provided by Goodfellow Metals Ltd., was used as the cathodic substrate. Electroforming was carried out only on the side towards the anode, while the other side was protected with an insulating varnish, which was supplied by RS Ltd. The cathode was fixed to the TUFNOL jig, which is shown in Figure 3.19.

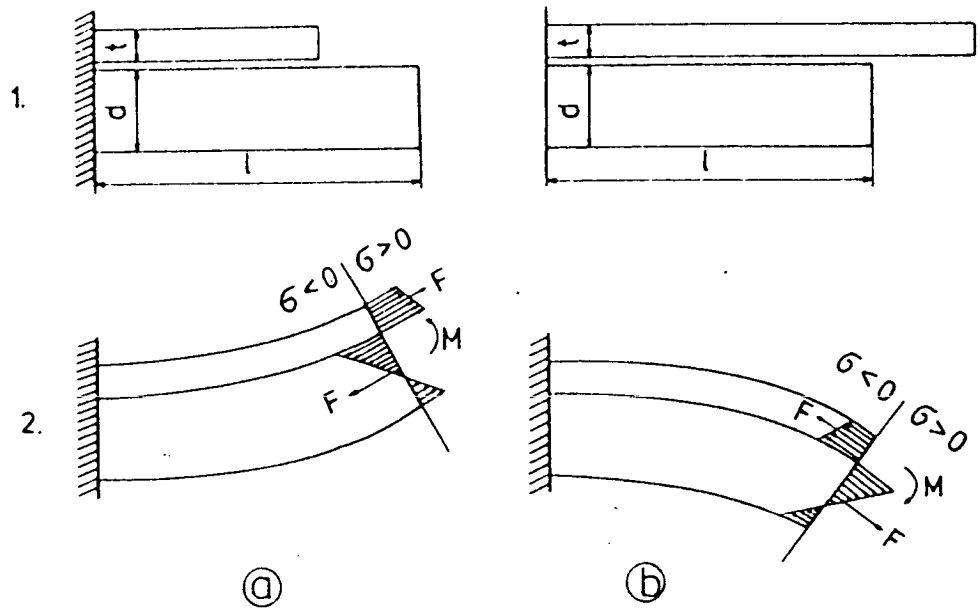


Figure 3.18 **Strained State of the Substrate Coating System During the Bent Strip Measurement: 1. the coating is imagined as being separated from the substrate; 2. elastic interaction between substrate and coating;** [48] **(a) tensile internal stress; (b) compressive internal stress**

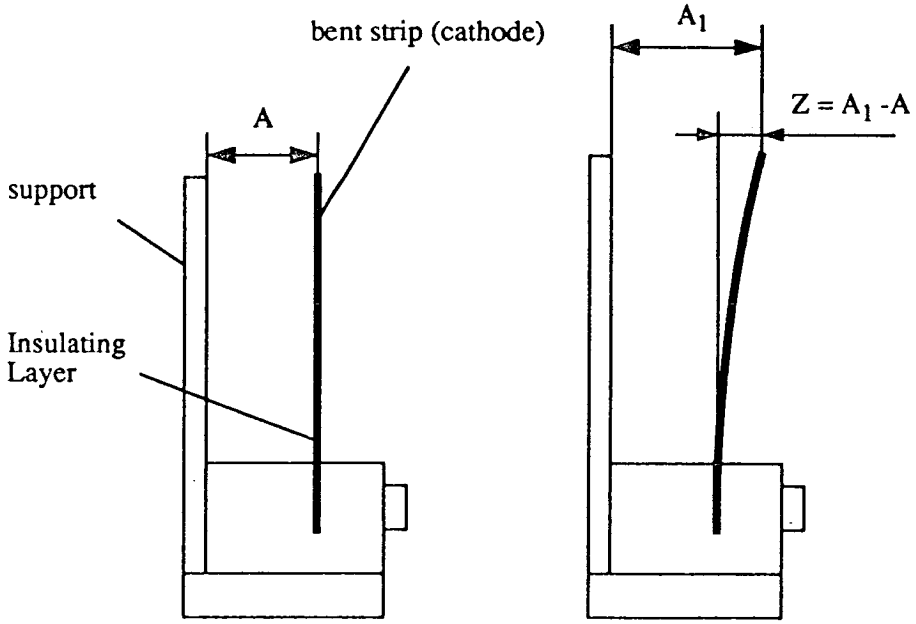


Figure 3.19 **Internal Stress Measurement by the Bent Strip Method**

The nickel anode is parallel to the cathode. Before electroforming, the free end gap A is measured by a microscope. Then the jig with brass substrate is put in the bath and current passed for one hour, and the gap A_1 was measured by microscope again. In the meantime, the coating thickness was also measured. When the coating thickness and the deviation between A and A_1 are obtained, the internal stress can be calculated by following formula[16,97]:

$$\sigma = \frac{1}{3} \times \frac{Ed^2Z}{tL^2} \qquad (3\text{-}3)$$

where E is Young's modulus of brass (100 GPa); d is the thickness of the brass cathode(0.2 mm); Z is the cathode deviation, t is the coating thickness; and L is the length of the brass(40 mm).

When the free end of the cathode has deviated from its initial position toward the anode, the internal stress is tensile. (Fig. 3.18 (a)). In the presence of compressive stress in the coating, the bending is in the opposite direction (Fig.3.18 (b)). The solution compositions and conditions are already given in Table 3.1. (section 3-2).

3-4-2. Effect of Deposition Variables on Internal Stress

I. Current Density

It is found that internal stress can be affected by current density. Most reports in the literature indicate that there is a rise in internal stress with increasing current density. Such results had been obtained in Watts,[22,48,92] and sulphamate

plating solutions[9,22,48]. In Figure 3.20 the relationship between the internal stress and current density are represented. Two different solutions were used for this study, via. 5% cobalt sulphamate and 3 ml/l EPC 30 agent with No3 bath at 50 ^{0}C temperature and pH 4. The two curves indicate that the internal stress increases at an even rate, as current density is increased. The results show that there are two directions of stress in the deposits. For the nickel cobalt sulphate solution, the stress increases in the tensile direction. For the hard nickel solution, the stress increases in the compressive direction.

It can been seen that minor stress changes occur in the low current density region. Between 1 and 5 A/dm^2, the stress only increases 1 MPa for nickel cobalt and 1.8 MPa for hard nickel. Larger changes were found between 5 and 20 A/dm^2.

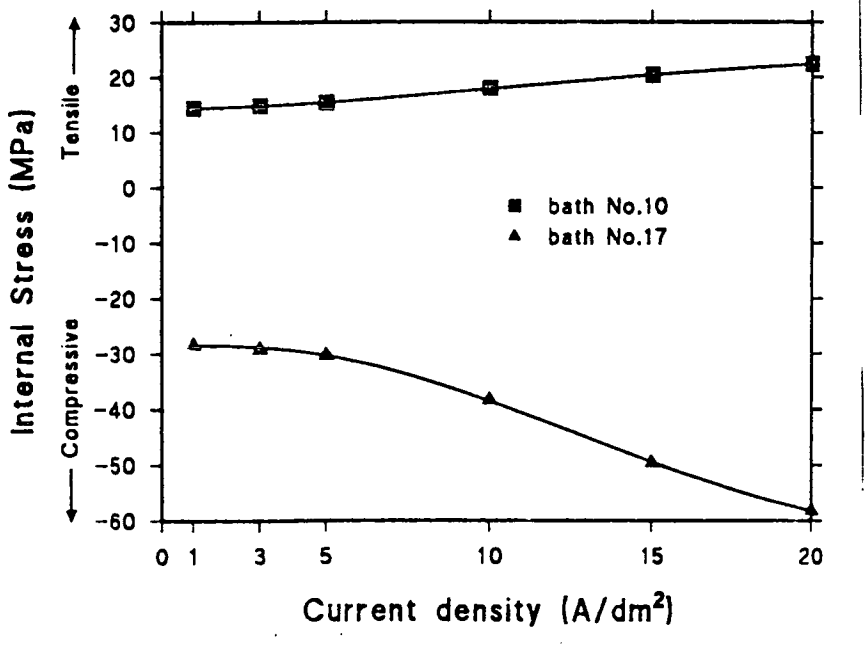


Figure 3.20 The Relationships Between the Current Density and Internal Stress at pH of 4.0 and 50 ^{0}C Temperature with Bath No.10 and No.17

II. Solution Composition

It is well known that the stress in nickel deposits is markedly affected by the composition of the plating solution. The chloride solution produces deposits with the highest internal tensile stress, and addition agents in some other solutions can produce deposits with the lowest internal tensile stress.[31] Marti [48] also reported that the increasing chloride content of a nickel bath tends to increase the tensile stress of the deposits.

In order to find the optimum solution to produce the lowest internal stress, three kinds of relationships were investigated. Figure 3.21 shows the dependence of internal stress on the content of nickel chloride in the solution. On the horizontal axis are marked the chloride content of the solution from zero to 100 g/l. The points on this curve were obtained by the internal stress values for 5 A/dm^2, and pH 4 value.The curve shows that the tensile stress increased

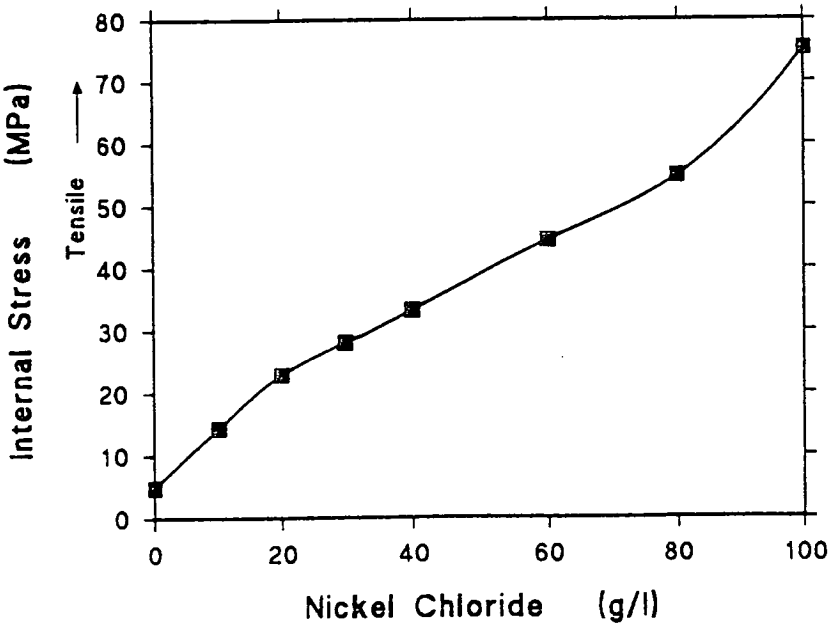


Figure 3.21 The Relationship Between Internal Stress and Nickel Chloride at 5 A/dm^2, pH of 4.0 and 50 ^{0}C Temperature with Nickel Sulphamate Bath

with increasing nickel chloride content of the solution. The internal stress reaches 75.2 ±3MPa at 100 g/l nickel chloride.

Figure 3.22 shows the relationship between cobalt sulphate concentration and deposit stress at a current density 5 A/dm^2 and solution temperature 50 ^{0}C. In this case, the lowest tensile stress appears at 5% cobalt sulphate content. From this point, the effect of increasing cobalt sulphate concentration is to increase the deposit stress in the tensile direction.

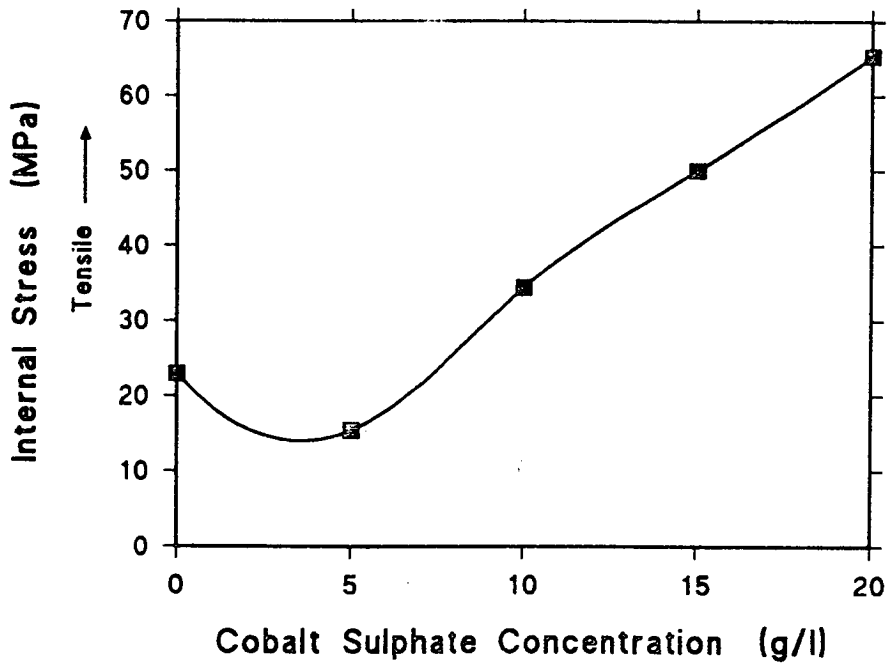


Figure 3.22 The Relationship Between Internal Stress and Cobalt Sulphate Concentration at 5 A/dm^2, pH of 4.0 and 50 ^{0}C Temperature with Nickel Cobalt Bath

The effect of EPC 30 agent upon internal stress is illustrated in Figure 3.23. The stress is changed from tensile to compressive with increase of EPC 30 concentration. However, the lowest internal stress can be obtained between the 0.5 ml/l to 1.0 ml/l EPC 30 agent with the basic nickel bath A. The compressive stress continues to increase at least up to at 5 ml/l EPC 30 concentration.

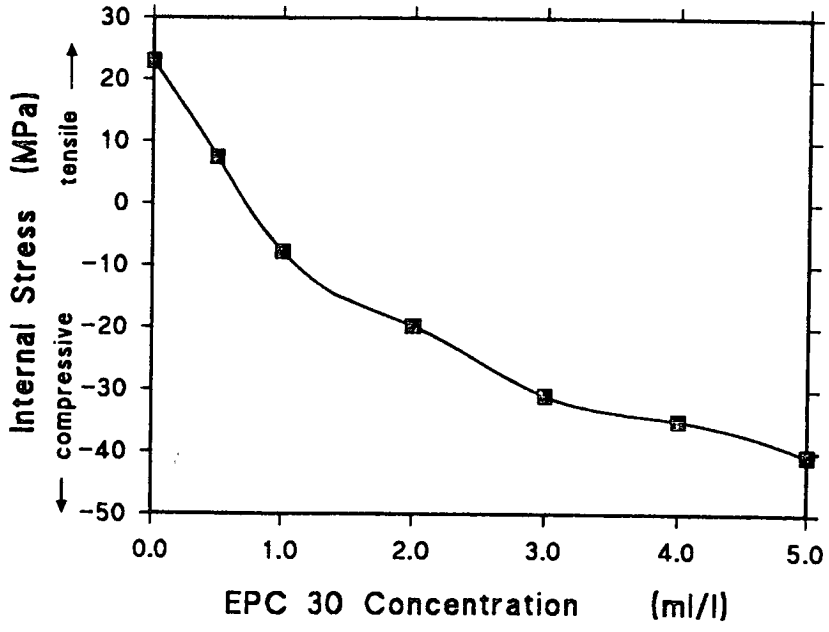


Figure 3.23 The Relationship Between Internal Stress and EPC 30 Concentration at 5 A/dm^2, pH of 4.0 and 50 ^{0}C Temperature with Hard Nickel Bath

3-5. MECHANICAL PROPERTIES OF ELECTROLESS NICKEL DEPOSITS

The word "electroless" is used here to denote a chemical plating process which resembles electroplating in that the process may be run continuously to build up a thick coating. Also, electroless plating can be succinctly described as a controlled autocatalytic chemical reduction process for depositing metals.

Interest in electroless plating processes has been growing rapidly in recent years, because electroless plating can be used for special applications for which conventional electrodeposition processes are unsuitable. For example, it can deposit metal uniformly on complicated shapes and down into recesses inaccessible to the flow of current. The nickel layer produced by the electroless process has high wear and corrosion resistance, high throwing power, high hardness and excellent uniformity. However, the electroless process has disadvantages. The process is slow, more expensive than conventional electroplating, and the nickel coating is quite brittle.

Is the electroless nickel layer suitable for the surfaces of dies and moulds? In order to understand its characteristics, mechanical properties of electroless are studied in this section. The flexibility and hardness were measured after the heat-treatment under different temperatures.

The simplest method for the determination of flexibility is a test based upon bending. The substrates are pieces aluminium sheet 50 mm long, 10 mm wide and 1 mm thick. The aluminium substrates were treated before the electroless process, the pretreatment process being shown in Table 3.2. Then, the samples were immediately immersed in the electroless nickel tank, (which is same one mentioned in section 2-5) for 20 hours after completing the final rinse.The

electroless solution was provided by M & T Chemicals Ltd. Birmingham, The composition of the bath and operating conditions are shown in Table 3.2.

NO.	SOLUTION	OPERATING CONDITIONS		
		Conc. (g/l)	Temp. (^{0}C)	Time (min.)
i	Non-Etch Soak Clean in M&T Alklean (A11)	30	60	5
ii	Mild Etch in M&T Alklean (A22)	50	70	3
iii	Alkaline Etch in M&T Alklean (77)	30	50	5
iv	M&T Alumseal 1000 Zincate Solution		30	2
v	Nitric Acid (HNO_3)	50	Room	1
vi	M&T Alumseal 1000 Zincate Solution		30	2

Table 3.2 Pretreatment Processes for Aluminium Alloy

The four edges of the coated samples were machined on a grinding machine until the aluminium substrates appeared. Then, the samples were immersed in 25% sodium hydroxide solution at a temperature of 80 ^{0}C to dissolve the aluminium sheet. Heat treatments were carried out in the furnace keeping one

hour for each sample at temperatures ranging from 100 ^{0}C to 700 ^{0}C. Hardness tests were made in a Vickers microhardness instrument with a load of 300 g. The hardness values were obtained by an average of five measurements. A JJ Lloyd M30 tensile test instrument was used to measure the load value and bending distance. The simple diagram is given in Figure 3.24. The instrument automatically recorded the load and bending distance at which the electroless nickel sample cracked. Therefore, the flexibility can be calculated from the following formula.[97]

$$D = \frac{4TS}{L^2} \ 100\ \% \qquad (3\text{-}4)$$

where: D: flexibility (%) T: Test sample's thickness (mm)

S; Bending distance (mm) L: Support span (30 mm)

The relationships of heat treatment to hardness and flexibility of electroless deposits are shown in Figure 3.25. It can be seen that the ductility decreases with increase of the hardness value. The hardness can reach nearly 1,070 ±10HV (300g) after the sample is heat treated at 400 ^{0}C for one hour. With the heat treatment at temperatures over 400 ^{0}C, the hardness decreases.

A similar result was reported by Gawne and Ma [30]. However, the layer of electroless nickel possesses very high hardness and high wear resistance. It is also found that the electroless nickel layer is very brittle and often not suitable for the surfaces of dies and moulds.

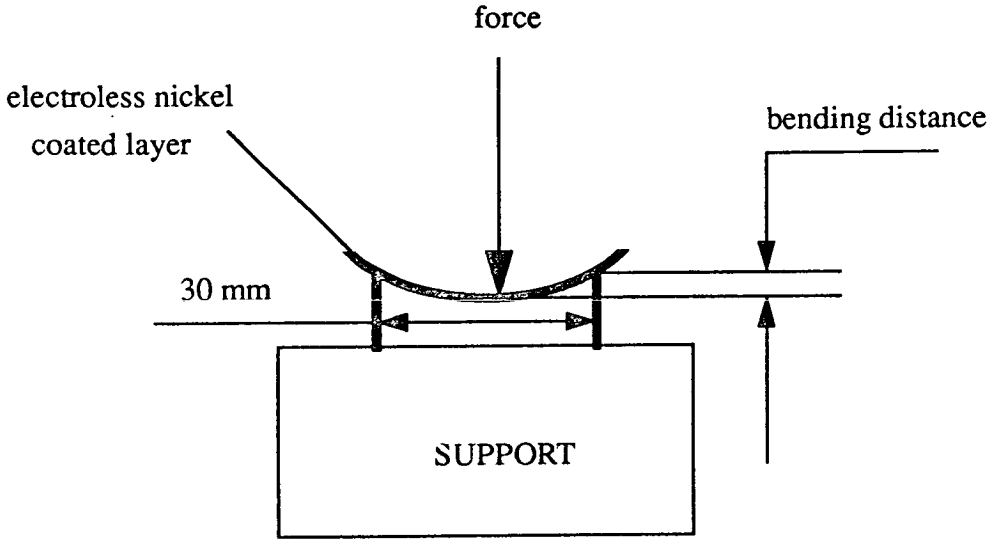


Figure 3.24 Diagram for Bending Test of Flexibility

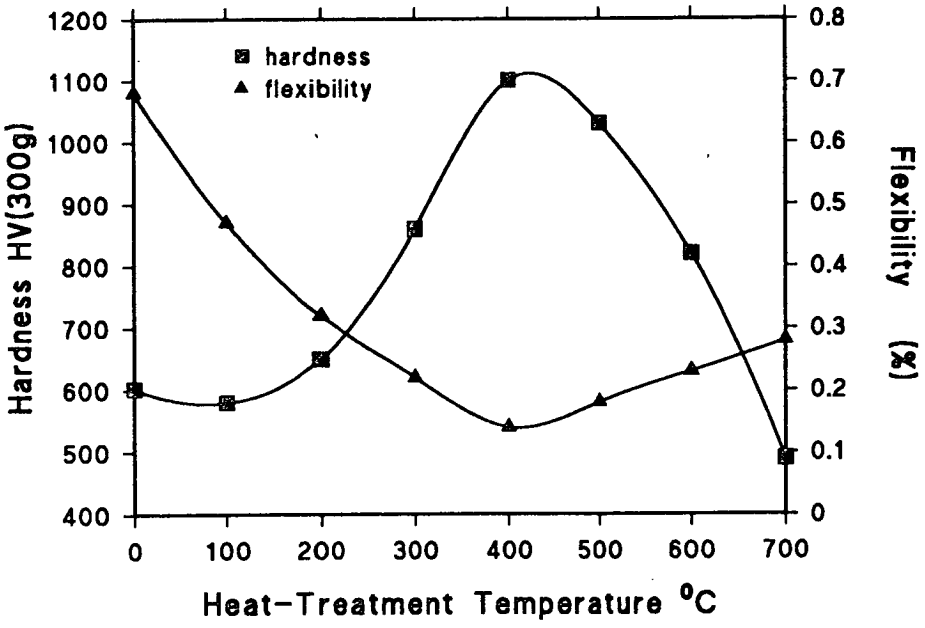


Figure 3.25 The relation of Heat Treatment to Hardness and Flexibility of Electroless Nickel Deposits

CHAPTER 4

PULSE PLATING

4-1. INTRODUCTION

Pulse plating has emerged as a new technique for deposition of metals and alloys. In conventional direct current electrodeposition, there is only one parameter, namely the current density, which can be varied. But in the pulse plating process, we have three main variables. They are pulse current density I_p, current "on time", and current "off time". The sum of "on" and "off" times constitute one pulse cycle. Some important relationships used in pulse plating are as follow:

$$DutyCycle = \frac{On\ Time}{On\ Time + Off\ Time} \times 100\ \% \qquad (4\text{-}1)$$

Average current density, $I_a = I_p$ x duty cycle. This density is similar to that used in D.C. electroplating. Duty cycles can vary from 1 to 100% and on time or off time from microseconds to milliseconds. So, in the pulse plating

we have an unlimited number of combinations of different pulse current densities for obtaining a given average current density. Simple square wave pulses are generally used for plating operations, though a variety of combinations of square wave pulses could also be employed.

Pulse plating is useful in certain cases where improved characteristics of electrodeposits are desired. Waveform, frequency and duty cycle of the pulsed current can affect the mechanical properties and internal stresses of nickel deposits. By pulsing the plating current, it is possible to exercise greater control. Some researchers[20,59,64] reported improvement in microthrowing power, hardness and reduction in porosity of nickel deposits with pulse plating. Celis and Roos [10] also reported that an improvement in brightness, crack free coatings and ductile deposits were obtained for nickel deposits by using the periodic reverse current technique. Corrosion resistance, brightness and surface roughness of Ni-Cu alloy deposits are improved by pulse plating[57]. Cherkaoui et al [14] found that pulse parameters can change the hardness value from HV 330 to 590, roughness R from 0.5 to 12 μm and internal stress from 6 to 20 kg/mm^2 in Ni-Cu deposits. They[15] also reported that the composition of the deposit was found to depend mainly on the average current density but only weakly on the pulse frequency and the pulse current density. Chen[12] has shown that very high instantaneous mass transfer rates corresponding to a current density of 100 - 1,000 A/dm^2 can be attained and that pulsing can change the current-potential relationships and as well as the morphology of the deposits.

In order to understand pulse plating characteristics, a special pulse power supply had been designed for the investigation of the effect of pulse plating on mechanical properties and internal stress. The influence of pulse parameters on

the electroforming shell dimensional accuracy is also studied.

4-2. PULSE GENERATION

Pulse generation plays an important part in electrodeposition process. Compared with a D.C. power supply, pulse generation is more complex. At present, pulse plating is generally performed with any of four generic waveforms; i.e., square, asymmetric sine, split sine and periodic reverse square wave. The four typical waveforms are shown in Figure 4.1.[65] Many variations of these basic waveforms can be used. For example, duplex pulse, pulsed pulse, pulse on pulse, pulse reverse, pulsed pulse reverse and pulse on pulse reverse, which are shown in Figure 4.2,[20] each with its own advantage and disadvantage. Square waves (Fig.4.1a) can be generated at very high frequencies (to 20 kHz). Periodic reverse square wave (Fig.4.1d) offers the advantage of catholyte metal concentration replenishment when the cathode is an anode for a short period of time. This can be disadvantageous in that dissolution of deposited metal can occur, inducing pits or cavities in the coating, but it can more often contribute to smoothing of the electrodeposit by removing asperities. This same statement can be made for asymmetric sine waves(Fig.4.1b), which can only operate at low frequency (50 to 60 Hz), and which are the least expensive. The split sine wave system (Fig.4.1c) produces current flow which is always cathodic and is generally used where the electroplated metal would dissolve in the electrolyte matrix.

Although more complex waveforms can be produced and are being utilized,(Fig.4.2) the square wave is the form generally accepted as the standard. In these experiments, square waveform was chosen, because it is superior to D.C.

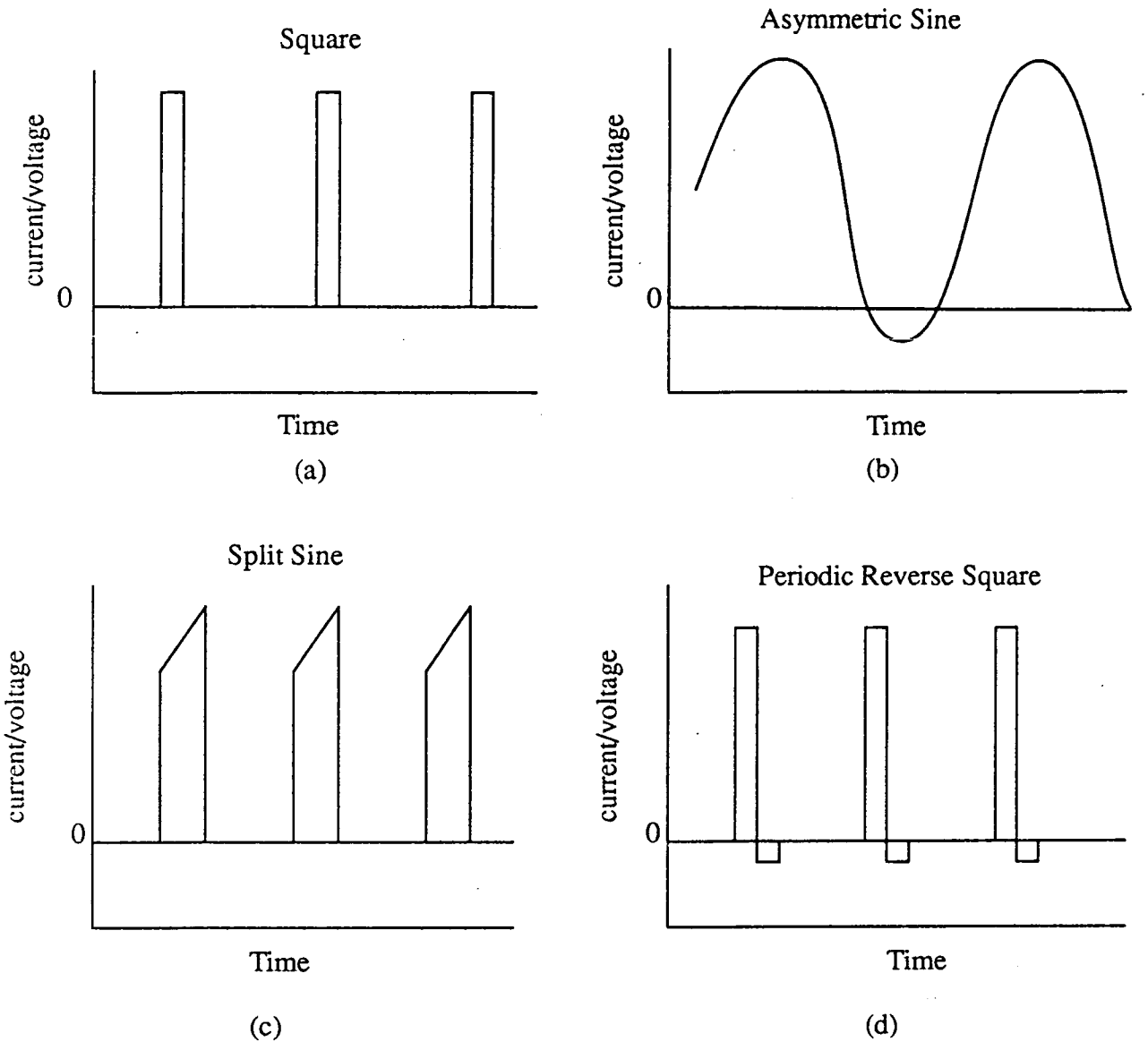


Figure 4.1 Four Generic Waveforms [65]

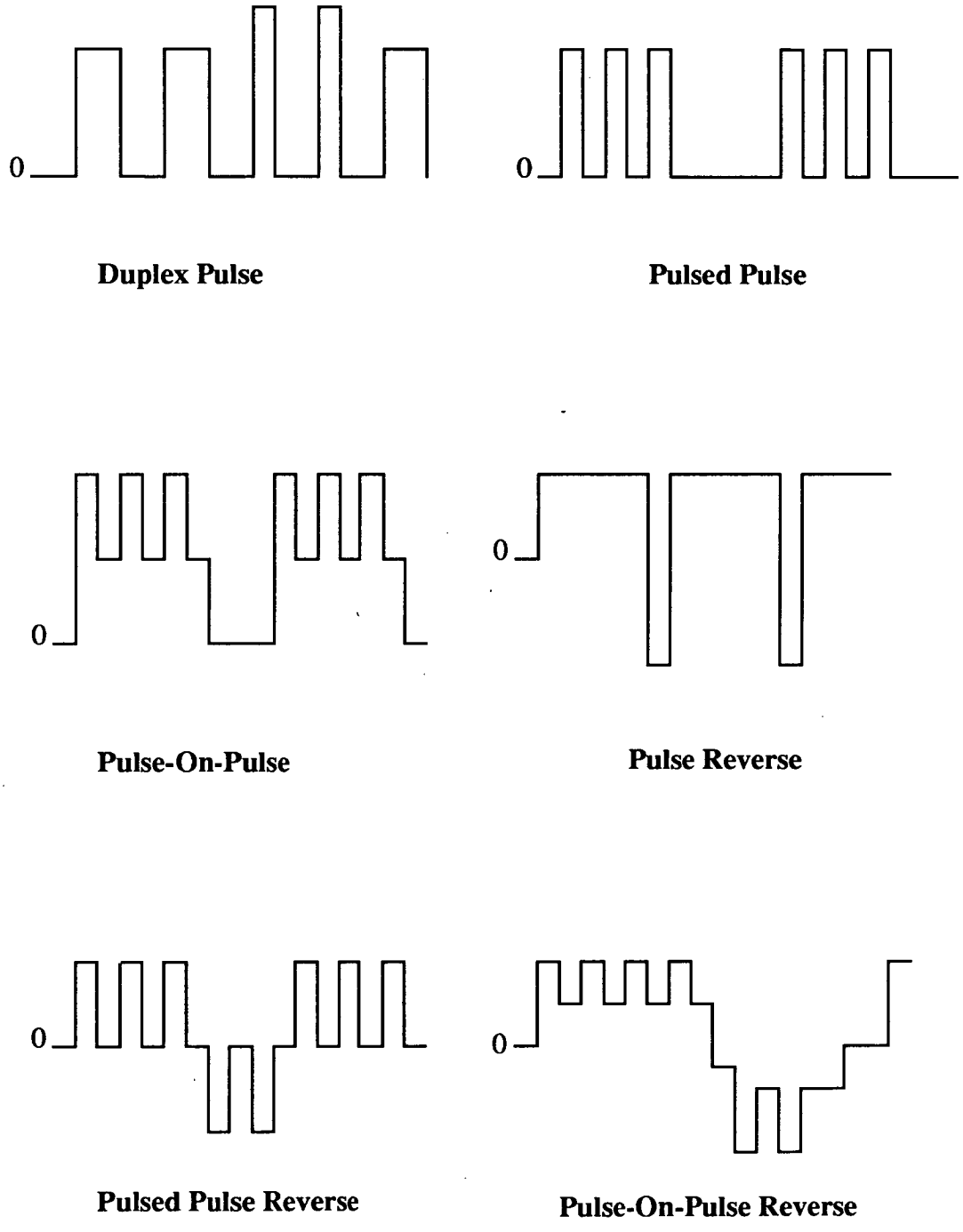


Figure 4.2 Different Waveforms of Pulse [20]

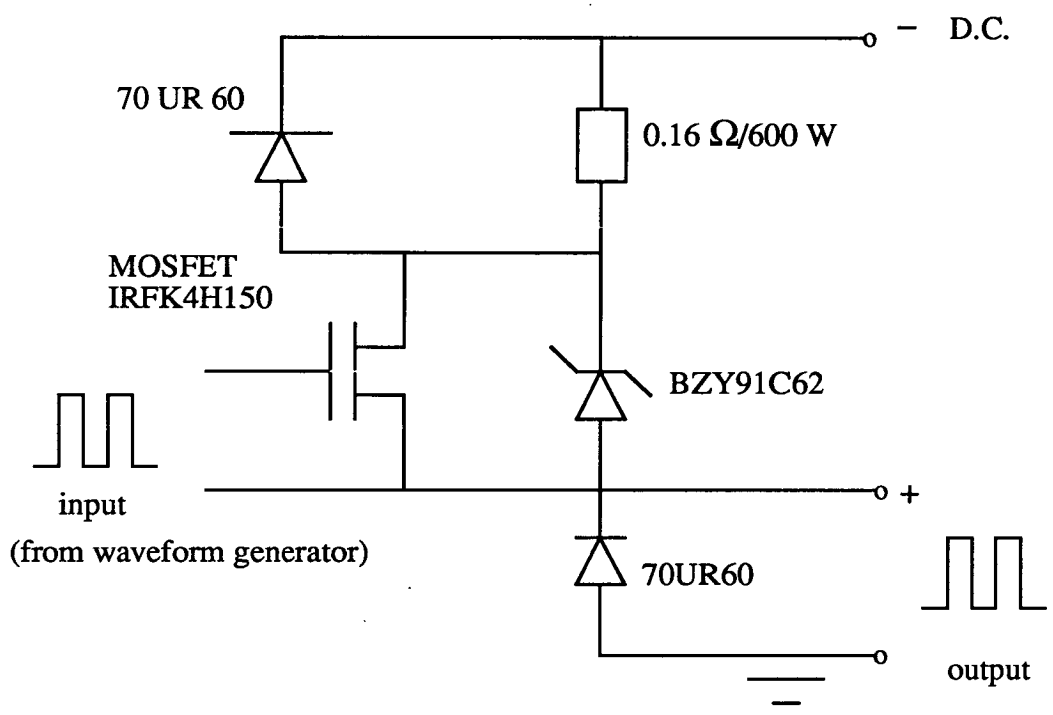


Figure 4.3 The Circuit Diagram of Pulse Power Supply

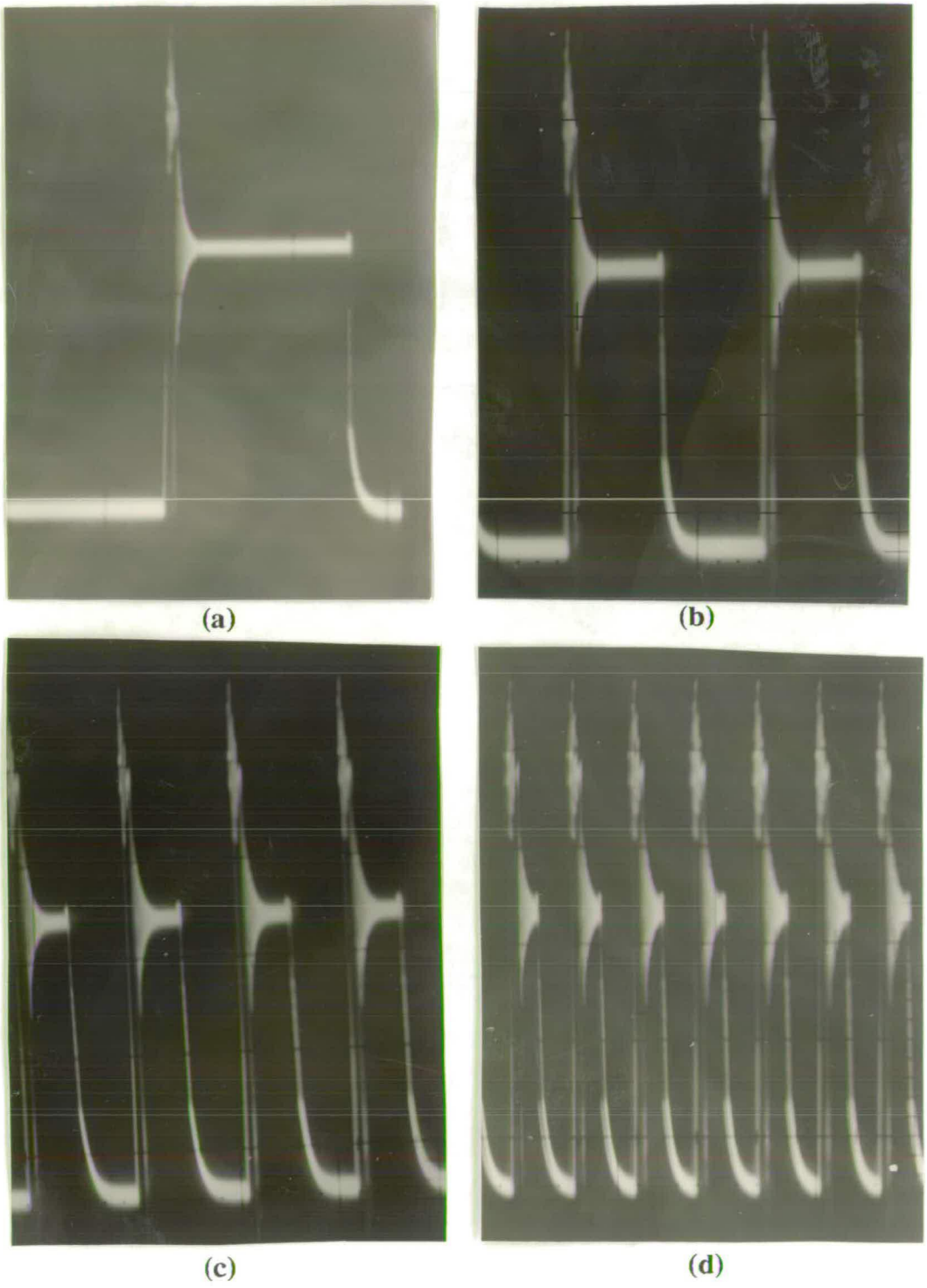


Figure 4.4 Working Waveforms With 50% Duty Cycle

(a) 5 KHz Frequency (b) 6.6 KHz Frequency

(c) 10 KHz Frequency (d) 20 KHz Frequency

power supply. The circuit diagram is shown in Figure 4.3. The waveform generator is provided by RS Ltd, (stock No.435-226), and the MOSFET is supplied by International Rectifier Co. (VDS = 100 V, TD = 137 A). This generator can produce pulsed square outputs from 20 Hz to 20 KHz without the need to switch range. The duty cycle can also be adjusted separately from 30% to 70%. The working waveforms are shown in Figure 4.4 [(a) 20KHz, (b) 10 KHz, (c) 6.6 KHz, (d) 5 KHz]. In this case, the duty cycle is 50% and every current density is 5 A/dm^2. It can be seen that it is not a "pure" square wave. The spike occurs in the end of peak current/voltage. (Sometimes, it can be as high as ten times the peak current/voltage). In addition, when spiking is seen, a reverse spike also appears on the down side of the wave. This back spiking changes the cathode to an anode for a short time at high current/voltage permitting unwanted electrochemical reactions to occur which might be either detrimental or beneficial to the electrodeposit.

4-3. PULSE PLATING EFFECTS ON MECHANICAL PROPERTIES

It is well known that the mechanical properties of nickel deposits can be affected by many factors, such as bath composition, temperature and current density, as discussed in Chapter 3. However, the pulse plating parameter, such as pulse current density (I_p), pulse frequencies and duty cycle can also influence these properties. Some researchers[14,35,55,80,81] have reported that change of pulse plating parameters can increase tensile stress, and decrease the internal stress, and yield a fine grained deposit. In order further to understand the relationships between the mechanical properties of nickel deposits and pulse plating, the following experiments were carried out; tensile stress, elongation,

hardness and internal stress in different pulse plating conditions with bath No.3 solution. The pH of the plating solution was 4.0 and its temperature was 50 ^{0}C. In all instances samples were deposited for comparison at the same unpulsed current densities, the average current density being held at 5 A/dm^2.

The samples for tensile stress were produced by the method, described in section 3-2. The procedures for measurement of tensile stress, elongation and hardness were also as before (section 3-2). 50% duty cycle, 10 A/dm^2 pulse current density and frequencies from 2.5 kHz to 20 kHz were chosen as pulse plating parameters. The variation of tensile strength and elongation with increasing pulse frequency is shown in Figure 4.5. The tensile strength increased with increasing pulse frequency and almost constant average current density. The elongation correspondingly decreased with increasing pulse frequency.

The relationship between the hardness and the pulse frequency is depicted in Figure 4.6. It can be seen that the hardness also increases with the increasing frequency. Some researchers[40] obtained the same results and believed that the effect of frequency on the grain size corresponded to that on the strength, the grain size decreased with increasing frequency. The overpotential also increased with increasing frequency and was apparently responsible for the grain refinement.

It was also found that the duration of the on-time of the pulse can affect the nickel strength. The relationship between the ultimate stress and on-time of pulse is shown in Figure 4.7. During this study, the pulse current density and off-time were kept constant, (10 A/dm^2 and 100 μs). On-time and average current density were varied. Figure 4.8 shows the scheme for investigating changes in on-time. The on-time ranged from 100 μs to 400 μs. Clearly, when the on-time increases,

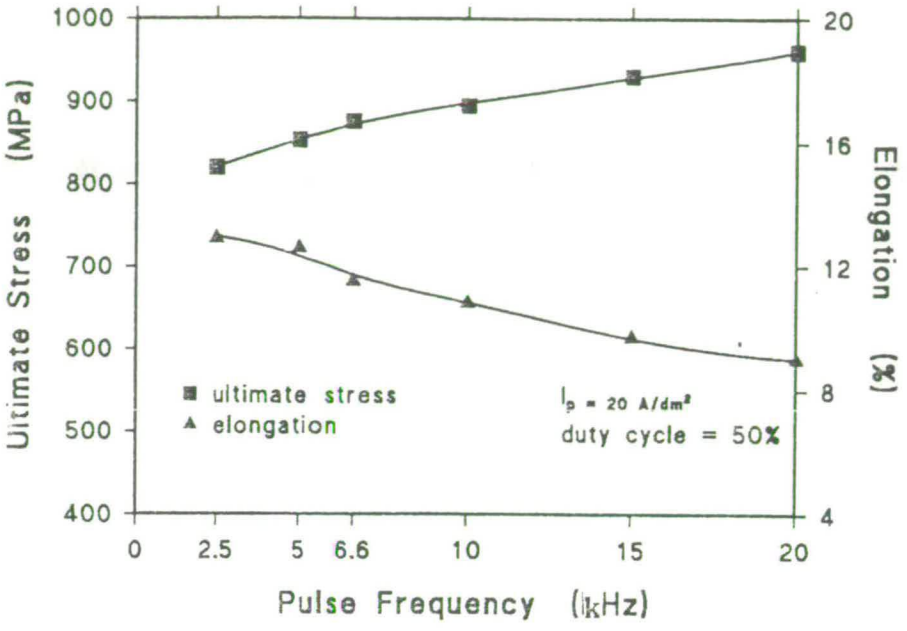


Figure 4.5 Variation of Ultimate Stress and Elongation with Pulse Frequency

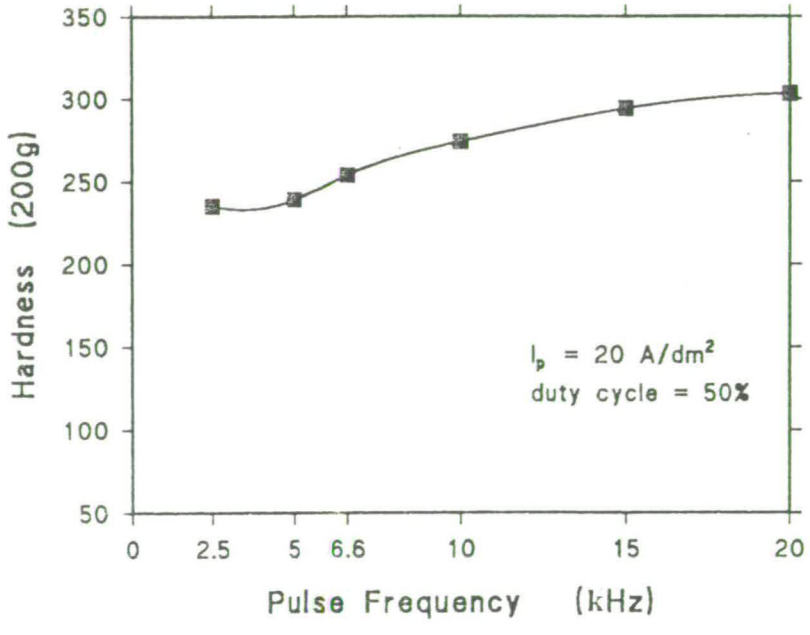


Figure 4.6 Variation of Hardness with Pulse Frequency

the average current density also increases. The result from Figure 4.7 shows that the ultimate stress decreased slightly with increasing on-time duration.

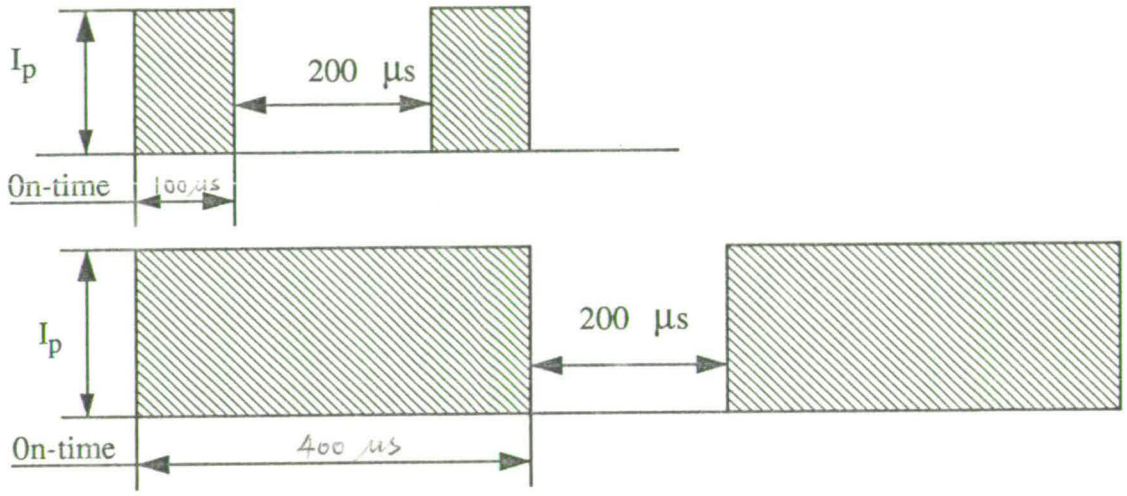


Figure 4.7 Scheme for Investigating in On-Time Duration

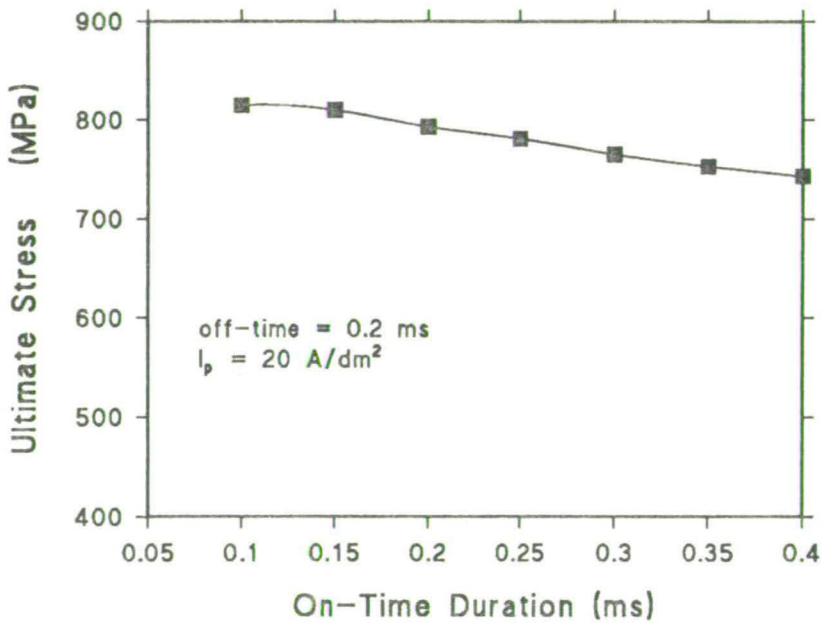


Figure 4.8 The Relationship Between Ultimate Stress and On-time Duration

Pulse plating parameters can also affect on the internal stress. In order to measure internal stress, the same experimental procedure was applied, and is described in section 3-4-1. No. 3 bath was used for these experiments. The relationship between internal stress and pulse plating frequency is shown in Figure 4.9. The pulse current density was 20 A/dm^2 and duty cycle 50%. The frequency was varied from 2.5 kHz to 20 kHz. It was found that the internal stress decreases with increasing pulse frequencies. This result is quite similar with Kim and Weil's result.[40] They believed that more hydrogen codeposited and remained in the layer when the pulse frequency was high so that there was less contraction. Thus the higher the pulse frequency, the more on-time beginnings there are per unit time. However, because the hydrogen remained in the layer because of its small diffusion coefficient, there would be less contraction and a lower tensile stress.

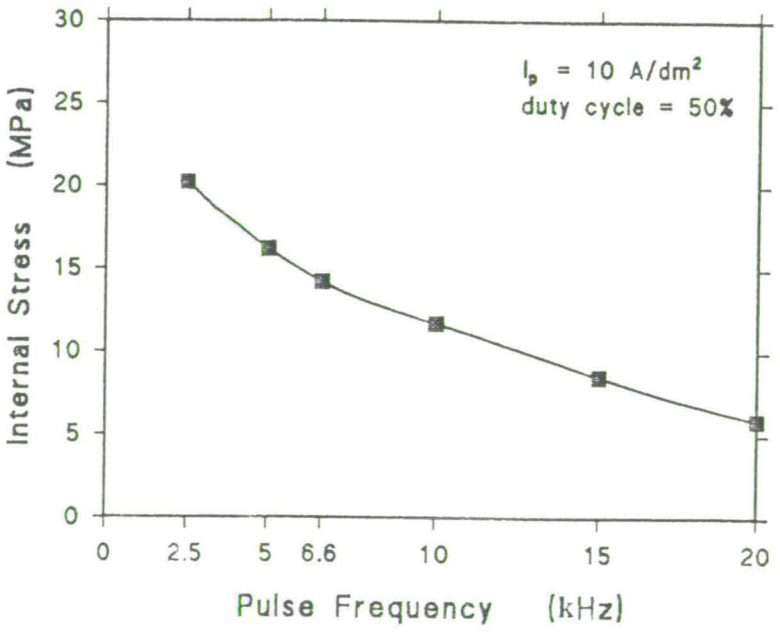


Figure 4.9 The Relationship Between Internal Stress and Pulse Frequencies

The variation in internal stress with on-time duration is depicted in Figure 4.10. During this study, the pulse off-time was kept constant, (200 μs). It can be seen that the internal stress gradually increases with increasing on-time duration. In this case, increasing on-time duration results in increasing average current density and decreasing pulse frequency. Both factors tend to increase internal stress.

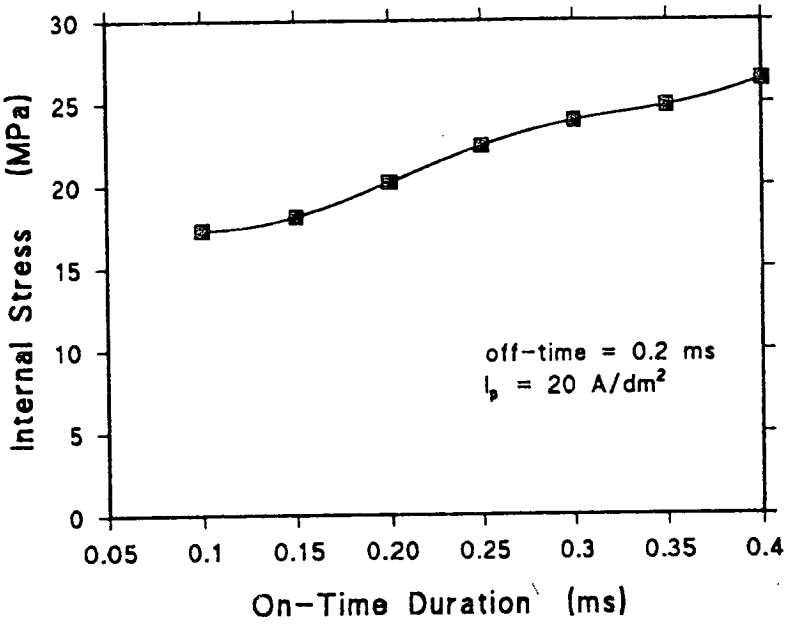


Figure 4.10 The Relationship Between Internal Stress and On-Time Duration

Pulse off-time also influences internal stress. In order to observe this phenomenon, the on-time duration and pulse current density were kept constant, (200 μs and 20 A/dm^2). The off-time interval ranged from 100 μs to 400 μs. The result (shown in Figure 4.11) shows the internal stress decreases with increasing off-time interval.

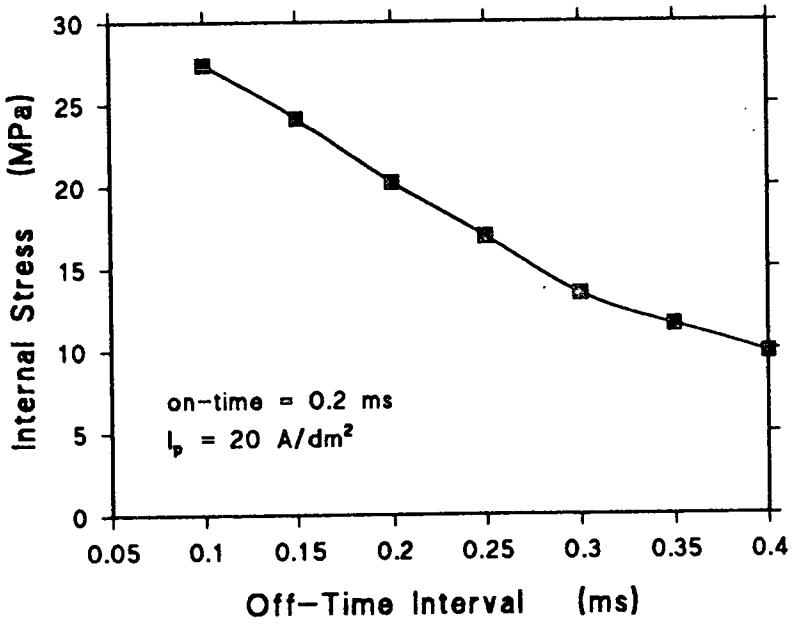


Figure 4.11 The Relationship Between Internal Stress and Off-Time Interval

4-4. INFLUENCE OF PULSE PLATING PARAMETERS ON THE ELECTROFORMING SHELL ACCURACY

It is well known that pulse parameters can change the mechanical properties of nickel deposit, and improve coating uniformity. Because pulse plating is a high frequency cycle in contrast to D.C. plating, there is less chance for high or low current density effects to be noted over the part being plated. These high or low current density effects can be related to ionic concentration differences across the part. It was stated that the equilibrium that can be obtained utilizing pulse plating to maintain uniform ionic concentrations across the surface of the part with the solution will promote good metal distribution as long as the pulsed cycle does not starve the catholyte and produce a capacitance effect.[26]

In order to analyse the effect on the electroforming shell accuracy of using pulse plating, the following experiment was carried out. The three steps sample was made of aluminium alloy and the dimensions are designated in Figure 4.12. Silicone rubber is used to produce a reverse mandrel as illustrated in Figure 4.13, and the nickel shell electroformed on it. The stepped shape was chosen to assist with measuring the dimensions by means of micrometer.

Since the silicone rubber is an insulator, the surface of the mandrel has to be made conducting, and fine graphite power was applied to the working surface to achieve this. No. 3 bath was used for these experiments. The average current density was kept at 3 A/dm^2.

In order to observe the effect of different plating conditions, four methods were chosen: D.C; and pulse plating at 5, 10, and 20 kHz frequencies with a duty cycle of 50%. The same rubber mandrel was used for all four plating methods. After 22 hours plating in each case four nickel replica shells were produced. A

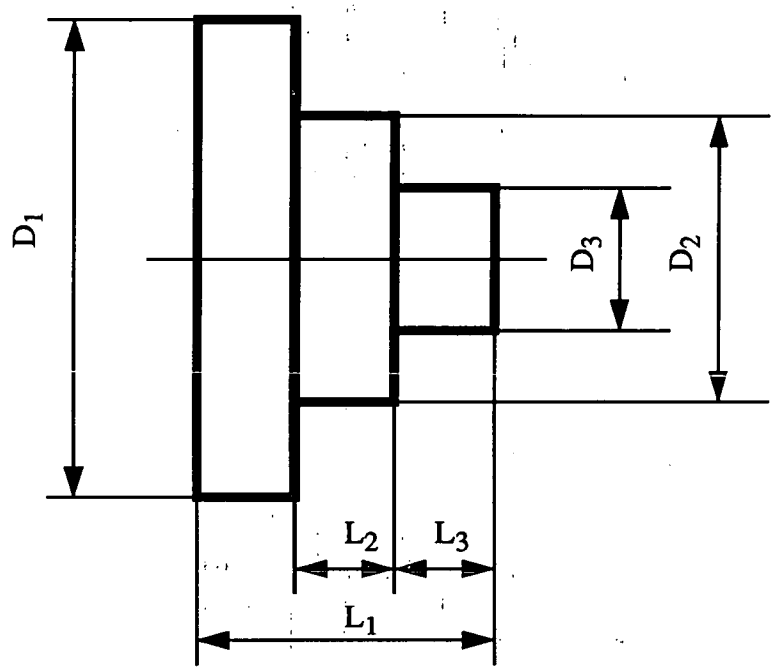


Figure 4.12 All Dimensions for Mandrel

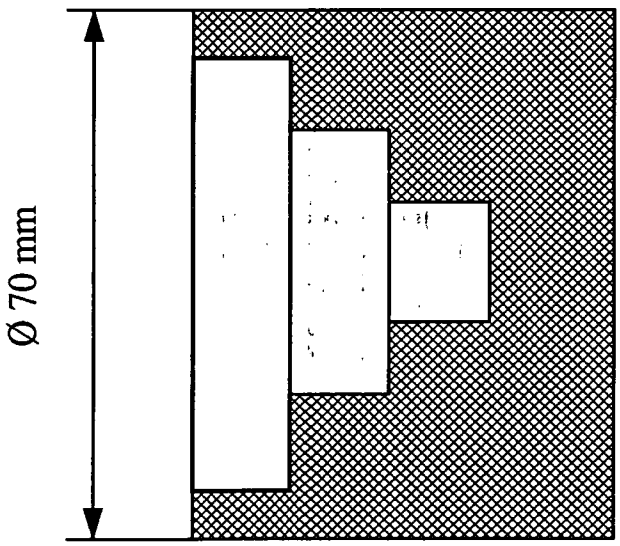


Figure 4.13 Reverse Mandrel of Silicone Rubber

Table 4.1 Data of the Mandrel and Nickel Shells

	D_1(mm)	D_2(mm)	D_3(mm)	L_1(mm)	L_2(mm)	L_3(mm)
Al. Mandrel	60.00	40.00	20.00	30.00	20.00	10.00
Ni. Shell by D.C.	59.63	39.60	19.70	29.65	19.80	9.90
Ni Shell by 5 kHz	59.80	39.75	19.85	29.80	19.91	9.92
Ni Shell by 10 kHz	59.81	39.80	19.90	29.82	19.93	9.93
Ni. Shell by 20 kHz	59.82	39.84	19.92	29.84	19.95	9.95

* all dimensions ±0.01

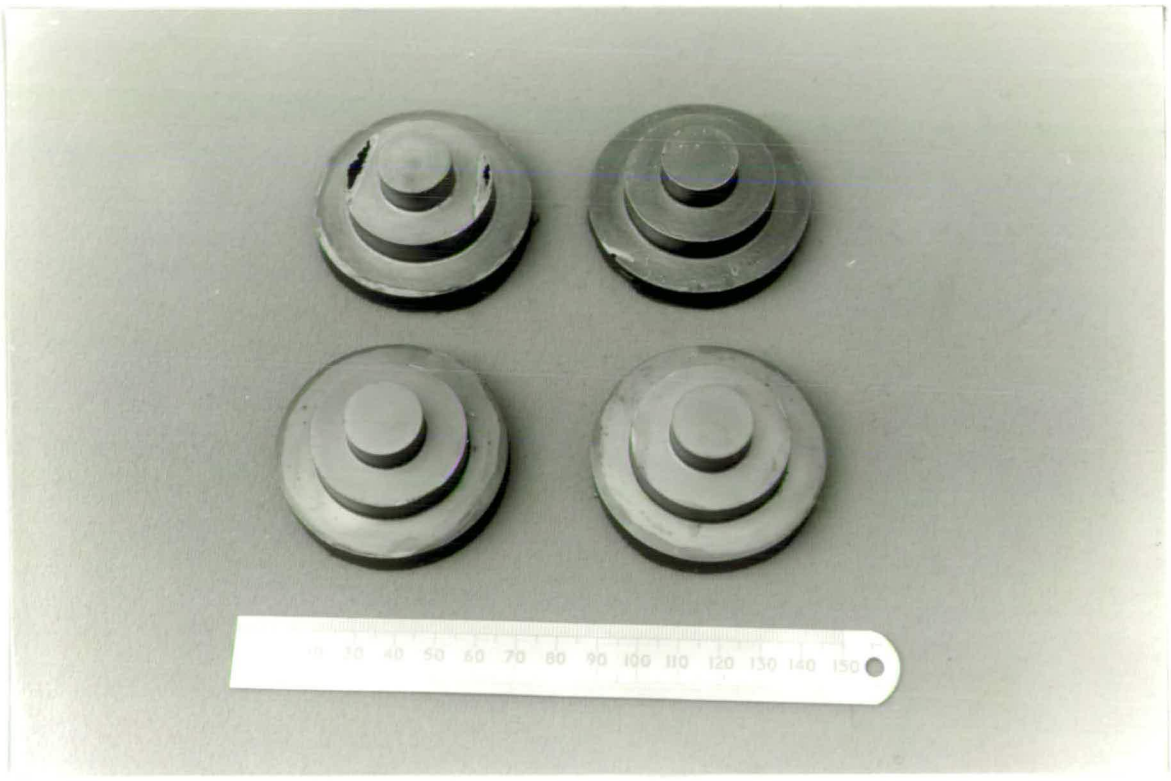


Figure 4.14 Four Nickel Shells (left upper by D.C; right upper by pulse 5 kHz left bottom by pulse10 kHz, right bottom by pulse 20 kHz)

micrometer was used to measure all dimensions. Results are shown in Table 4.1.

Figure 4.14 is a photograph of the four nickel shells. It was found that the nickel shell produced by D.C. had a big crack in first step surface at the outer edge. With increasing pulse frequency, the nickel shell surface quality became better. In addition, the dimensional tolerances tend to be better, and the metal distribution over the shell being plated is more uniform.

These results demonstrate that pulse plating can not only improve the surface quality, but can increase the dimensional accuracy of the nickel shell. These results further confirm that internal stress decreases with increasing pulse frequency at least up to 20 kHz. During D.C. plating, a big crack in first step surface appeared, the main reason being internal stress. Because the mandrel was silicone rubber, the strength of adhesion between the rubber surface and nickel layer was small. When the internal stress was larger than the adhesive strength, the coated layer peeled off the rubber which caused the crack. With high pulse frequency, the crack disappeared.This advantage is very important for making dies and moulds.

CHAPTER 5

FORMERS AND PATTERNS

5-1. INTRODUCTION

The object of this part of the project is to investigate mandrel materials, their processing, their contours and the influence of these on the electroformed shell. In order to produce high quality dies and moulds by electroforming the process, the best mandrel materials and their preparation are vital to the success of the process. The surface preparation of mandrels is a critical step in producing an acceptable electroformed article, and is described in this chapter. In addition current (and therefore metal) distribution must normally be as uniform as possible and the ability to control it is the key to successful electroforming. The limitations of the electroforming process are studied. The throwing power is improved by various means. Finally, the accuracy of reproduction by electroforming is measured and analyzed.

5-2. CHOICE AND REMOVAL OF MANDREL

The choice of material for the mandrel is very important and has involved a wide ranging research operation which is still continuing. Usually, mandrels may be classified as conductors or nonconductors of electricity, and each of these may be permanent, semi-permanent or expendable. Whether or not a mandrel is a conductor will determine the procedures required to prepare it for electroforming. Conductive mandrels are usually pure metals or alloys of metals and are prepared by the usual procedures, but may require application of a thin parting film to facilitate separation of the electroform from the mandrel. Nonconductors must be made conductive by spraying the surface with a thin metallic film, usually silver or by other metallization techniques.

Whether or not a permanent or expendable mandrel should be used is largely dependent on the particular article that is to be electroformed. Most of the mandrels used for making dies and moulds are collapsible, or removable by chemical dissolution. Mandrel materials satisfying these requirements include polyvinyl chloride,[52,82] epoxy resin,[4,18] and aluminum.[3] Each mandrel material has its own advantages and disadvantages.

Stainless steel is an excellent material to use as a permanent, conductive mandrel. Although machining may be relatively difficult, it can be machined to close tolerances and given a high surface finish. As it is conductive, there is no need to metallize the surface before beginning the electroforming process. Stainless steels are preferred because they readily form stable, passive films that prevent adhesion between electroform and mandrel.

Expendable conductive mandrel materials include aluminum and zinc alloys, and low-melting materials. Aluminum or one of its alloys is used because it easy

Expendable conductive mandrel materials include aluminum and zinc alloys, and low-melting materials. Aluminum or one of its alloys is used because it easy to machine to close dimensional tolerances and can be given a high surface finish. However, it is relatively high in cost, and easily damaged; it does not always require metallization prior to electroforming. When used as an expendable mandrel, aluminum or its alloys can be dissolved away from a nickel electroform in 25% sodium hydroxide.

Nonconductive mandrel materials include wax, glass, silicone rubber and plastics. These materials are expendable or, at best semi-permanent.

Wax is useful because it can be readily cast and easily melted out of the finished electroform, but it has many disadvantages. It may be brittle, it has a poor surface finish and it is easily damaged. It is inexpensive, requires metallization and cannot be used to maintain tight tolerances.

Plastics mandrels can be used to fabricate permanent or semi-permanent mandrels, but being nonconductive, require the use of silver spraying or other metallization techniques. In general, plastics are cheaper,but have inferior mechanical properties compared with metal mandrels. It is also difficult to obtain close dimensional tolerances and good surface finish. Rigid plastics mandrels are often made from epoxy resins.

Silicone rubber is another mandrel material. It is often easier to duplicate the complicated textures and patterns from workpieces by casting. It is also easier to separate from finished electroformed shell. The surface has to be treated and made conductive by silver spraying or other coating processes. It cannot hold a tight dimensional tolerance due to its shrinkage.

In order to decide which kind of material is used for the mandrel in these experiments, the following materials were tried:

Metallic materials: Stainless steel and aluminum alloys

Non-metallic materials: Polyurethane, SLA (material used in stereolithography) and silicone rubber*.

* which is called SILASTIC J RTV, base with curing agent, from Dow Corning Europe

All the mandrels were provided by DTI (Danish Technological Institute). Detail dimensions of the mandrel selected are shown in Figure 5.1. There are four different surface finishes which were created by turning, milling, spark eroding and lapping.

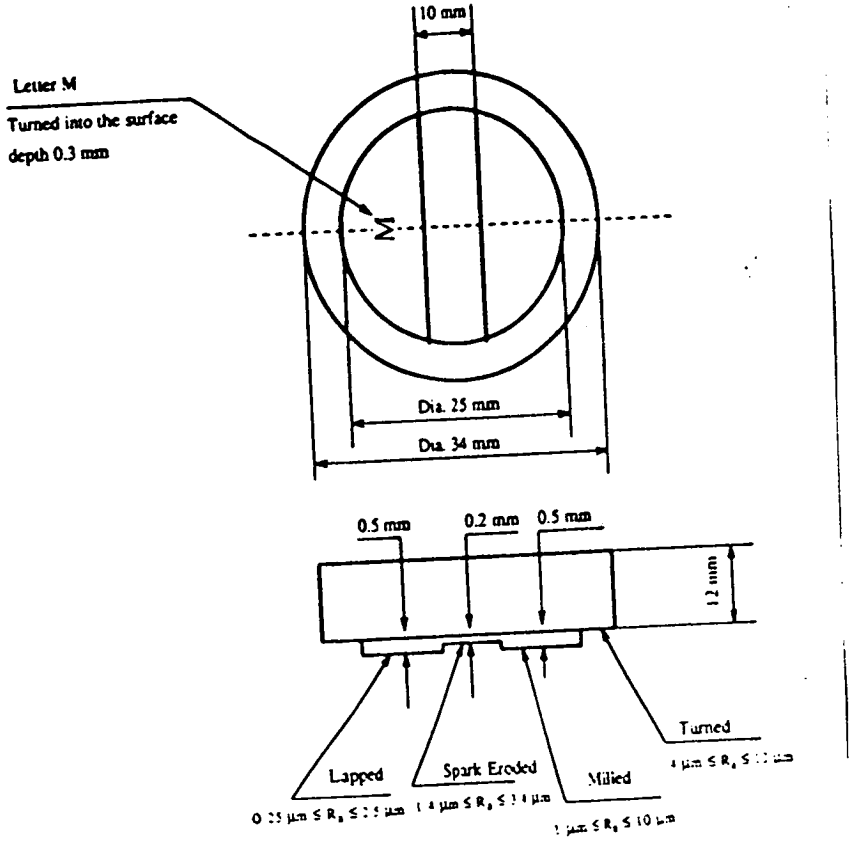


Figure 5.1 Dimensions of Aluminium Mandrel with M Letter

In order to produce a good nickel shell, the surface of the mandrel has to be treated by a series of chemical solutions. There are several processes, such as cleaning, etching, sensitising and activating.

For stainless steel, the surface is scrubbed with magnesium oxide, rinsed and passivated by immersion in a 2% solution of sodium dichromate for 30 to 60 seconds at room temperature. After this, this mandrel must be rinsed several times to remove all traces of the dichromate solution.

Aluminum alloys require special treatments when they are used as expendable mandrels to be removed from the electroform by chemical dissolution. The reason for this is the difficulty in obtaining sound electrodeposits directly on aluminum alloys from electroforming solutions. In this case, it may be necessary to use a proprietary zincate treatment to achieve a degree of adhesion that will prevent lifting and curling of the deposit from the mandrel. These special treatment processes are given in section 3-5, Table3.2.

Nonconducting mandrels must be made impervious to water and to the processing solutions, and then rendered conductive. Nonconducting materials may be rendered conductive in a number of ways.[18,49] Three methods were used in these experiments. The surface of the mandrel must be cleaned before applying the conductive film.

Firstly, a chemically reduced film of nickel was applied to the surface. In order to deposit the electroless nickel on the nonconducting mandrels, a sensitization process has to be carried out. The treatment processes are shown in Table 5.1. Then, the mandrel was immersed in electroless nickel bath No. 20 for 15 min. A very thin nickel film was deposited on the nonconductive material, (polyurethane, SLA, and silicone rubber). It was found that this process was not

suitable for a silicone rubber mandrel. After finishing the electroless nickel deposition, the silicone rubber was coated with quite a good nickel film, but when the coated rubber was taken from the hot electroless nickel bath at 90 ^{0}C to the nickel electroforming solution at 50 ^{0}C, the nickel film peeled off the silicone rubber, and sometimes, produced wrinkles which made the nickel shell unusable. These failures are attributed to the large thermal expansion differential between the rubber and the nickel due to the rapid temperature change from 90 ^{0}C to 50 ^{0}C.

No.	SOLUTION	OPERATING CONDITIONS		
		Conc.(g/l)	Temp.(^{0}C)	Time (min.)
1	Ammonium Fluoride Sodium Chloride	100 100	60	15
2	Stannous Chloride ($SnCl_2 \cdot 2HO$)	30	40	5
3	Palladium (P_dCl_2)	0.1	40	5

Table 5.1 Treatment Processes for Nonconducting Mandrels

The second method of making the silicone rubber mandrel conductive is by use of fine graphite powder. The powder was applied to all mandrel surfaces with a fine brush. This process makes the rubber conductive. Occasionally, gaps were found in the finished nickel shell. In order to avoid this drawback, the final process involved spraying the mandrel with silver. A spray gun was supplied by RS Co. (stock No.672-671). Electrodag 915 High Conductivity Paint, provided by Acheson Colloids Co., was used as the silver suspension after dilution with 4-methylpentan-2-one.

After preparation of the surface, all mandrels were electroformed in No.3 bath for ten hours. It was found that some materials, such as stainless steel, and aluminum, were very difficult to separate from the electroform.

After consideration of these results, two mandrel materials were chosen. One is the aluminum because it is quite soft and easily machined. On the other hand, no matter how complex the shape, the mandrel can be dissolved in 25% sodium hydroxide. The other material is silicone rubber, which is cheaper because no machining is involved and can be used again; also it is easy to replicate a complex pattern by the casting process. For example, if we have a plastic toy sample, we can use the silicone rubber to copy twice and reproduce the original in rubber. The four stages of the process are shown in Figure 5.2. The top left is the original plastic toy. The top right is the negative silicone rubber mould which is copied from the toy. The bottom left is the positive silicone rubber mandrel, which is cast from the silicone rubber negative. The bottom right is the nickel shell, which was made from a silicone rubber positive. It can be seen from this photograph that the nickel shell had a good surface finish, Also the detail of the plastic toy was well replicated on the nickel shell. In addition, the silicone rubber mandrel was

very easy to remove from the nickel shell. Consequently, this mandrel can be used many times.

Figure 5.2 Stages of the Electroforming Process

5-3. INVESTIGATING THE EFFECT OF SURFACE GEOMETRY

It is well known that there are limitations in electroforming. For example, sharp angles, corners and very deep, narrow recesses may cause problems.[58] Also it is difficult to achieve great or sudden changes in wall thickness by electroforming. The main reasons is that the current distribution is uneven and

causes non-uniform metal distribution on mandrel surface. Some researchers[24,36,72] found that modification of the current distribution can be achieved by changing the geometry of the part, by using auxiliary anodes or shields, or by changing the rack design. The following sections will discuss these problems in detail.

5-3-1. Analyzing the Influence of Current and Metal Distribution on the Nickel Shell

It is recognized that almost every practical plating arrangement results in a non-uniform current distribution. This is tolerable to a degree but often requires improvement to produce acceptable work. An important problem in metal distribution is deposition into recesses. Most parts have some hollows, angles and corners. A typical angle is shown in Figure 5.3.[63] Theory tells us that in a perfect angle, with perfectly sharp corners, the primary current density is infinite on the sharp external corners of the angle and zero on the corresponding internal corner. From Figure 5.3, it can be seen that the current flow is in straight lines, and that when the anode is farther away, the distances from the anode to near and far points of the angle are more nearly equal. However, the anode AB could be moved up as close as position CD without affecting the current distribution.

The best method to obtain even current distribution is that the anode should be the same shape as the mandrel. For instance, in plating into a large angle, which is shown in Figure 5.4a, the best anode arrangement is same angle. In most cases in industry, it is difficult to manufacture complicated anode shapes. Instead auxiliary shields may be used to improve the current distribution. Like Figure5.4b, two insulating plates were put between the anode and mandrel,

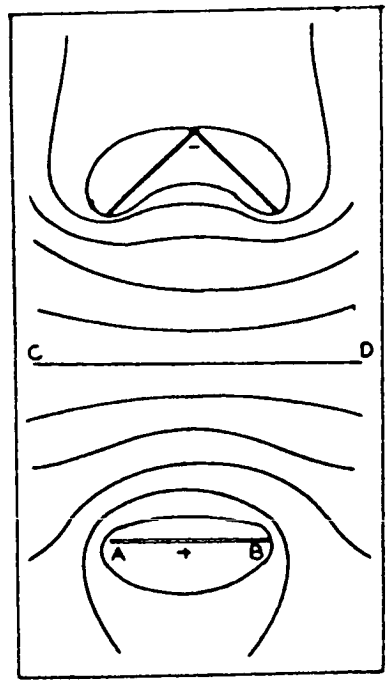


Figure 5.3 Equipotential Lines Between Plane Anode A-B and Angle Cathode in Bath [63]

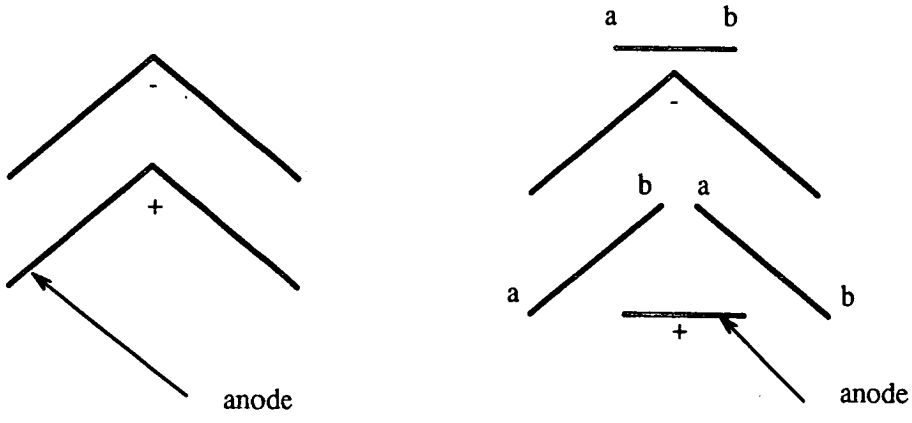


Figure 5.4 Angle Cathode with Various Arrangements (a) Conforming Angle (b) Insulating Plates

allowing current through the gap to the bottom of the angle cathode.

In order to confirm this phenomenon, an aluminum motor part with four deep holes, which was supplied by MAGMA GmbH Co. in Germany, was used as the mandrel to produce a nickel shell. The motor part is shown in Figure 5.5. After pretreatment, the mandrel was coated in nickel bath No.3 using a plane nickel anode. It was found the bottom of the four holes could not be completely coated even after 24 hours. Thick nickel was deposited at the edges of the holes because the current was concentrated there. In order to improve this case, a silicone rubber shield or mask was made to insulate the top surface, but leave the four holes exposed. Current can pass through the silicone rubber holes to deposit metal on the four holes of the motor part. The schematic is shown in Figure 5.6. After ten hours plating, the silicone rubber shield was taken off, then the whole motor part was electroplated. The result shows that a good nickel formed layer can be deposited on the bottom of the four deep holes.

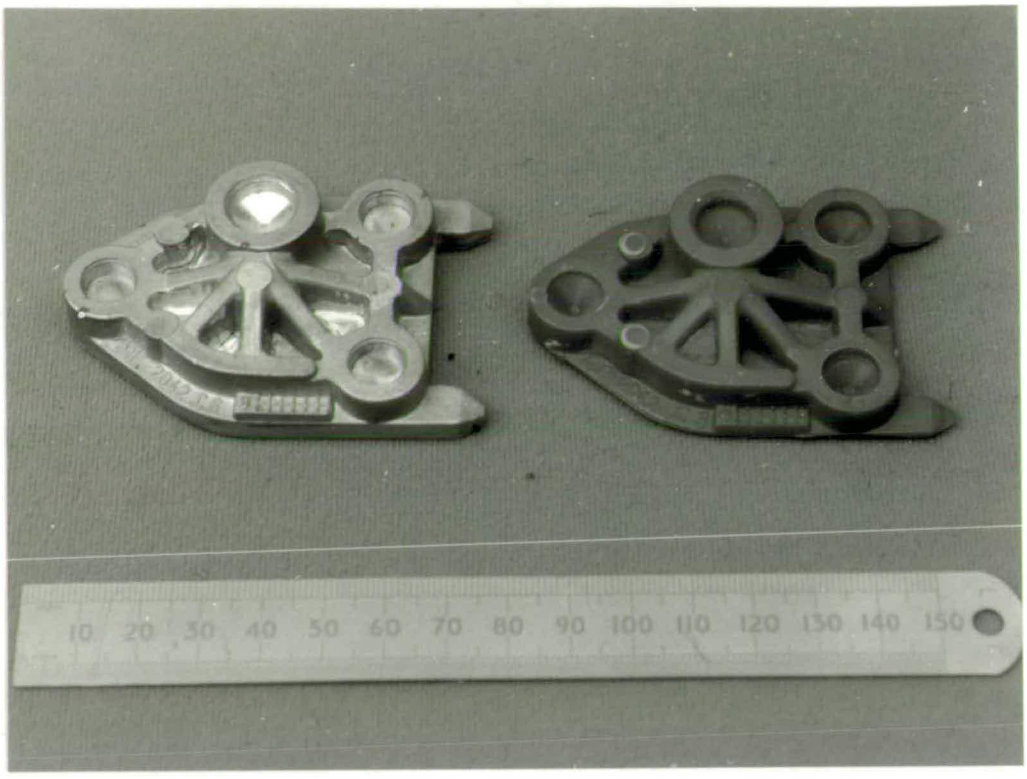


Figure 5.5 View of Aluminum Motor Part

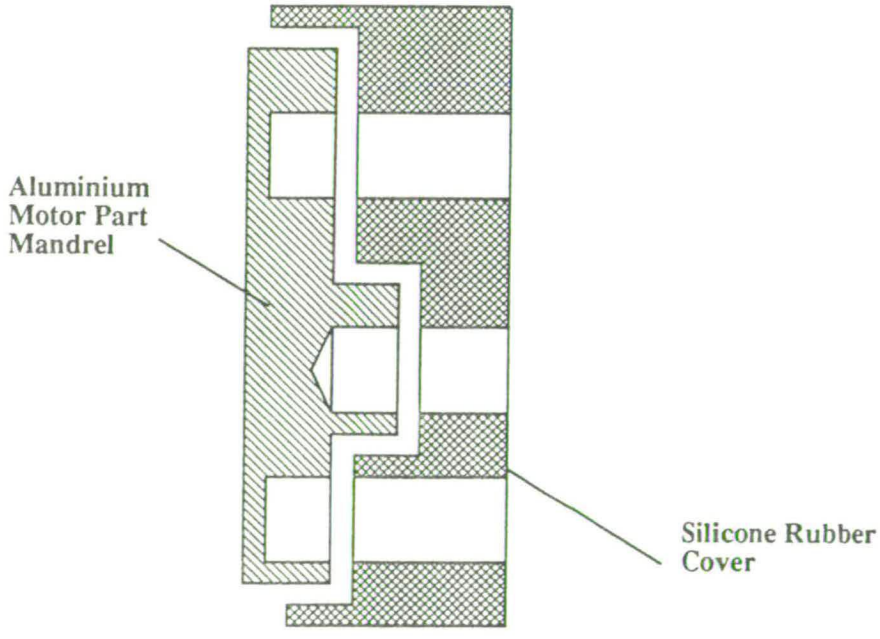


Figure 5.6 Schematic of Silicone Rubber Cover Used over Motor Part Mandrel

5-3-2. The Effect of Deposition Conditions on Throwing Power

Throwing power is an important consideration in practical electroforming processes and may be defined as the measure of the ability of a solution to deposit a uniform layer of metal on an irregular surface, in effect its ability to deposit metal in cavities. Throwing power is sometimes measured using a Hull Cell. Typical plating solutions include silver cyanide and chromium - based solutions with high and low throwing power respectively. Some researchers[61,75,86] found that modification of the pulse parameters and bath composition can improve throwing power.

In order to observe the extent of plating penetration under various conditions, four identical aluminum alloy parts were prepared. The dimensions are shown in Figure 5.7, each piece has 12 drilled holes of diameter 10 mm with different depths (from 2 mm to 24 mm). The aluminium parts were treated in various chemical solutions. The processes are shown in Table 3.2 (section 3-5). Two kinds of solutions were used, No.3 bath and electroless nickel No.20 bath. (see Table 3.1)

The back and sides of each test piece were insulated leaving the front bare. After depositing for ten hours, the coated parts were sectioned across each hole. Then the section surface was polished and etched in 5% HNO_3. The coating thickness at the base of the hole was measured every 2 mm across a diameter using a microhardness instrument to move the table and align the cross hairs in the eyepiece with the coated layer.

The results are shown in Table 5.2 to Table 5.4. The operating conditions are also described with them. For electroless nickel, an even coated layer was obtained. All thicknesses were about 100 μm. It can be seen in Figure 5.8 that

even at the bottom of a 20 mm deep recess, there was a uniform electroless nickel layer. Another three sectional samples are separately shown in Figure 5.9 to Figure 5.11.

To compare the different throwing effects, four sets of data were obtained using different coating conditions and the relationships between the depth and coated thickness are shown in Figure 5.12. It is very clear that electroless plating has the best throwing power. Pulse plating and the use of an insulating plate can also improve the penetration. The normal D.C. plating process has low throwing power.

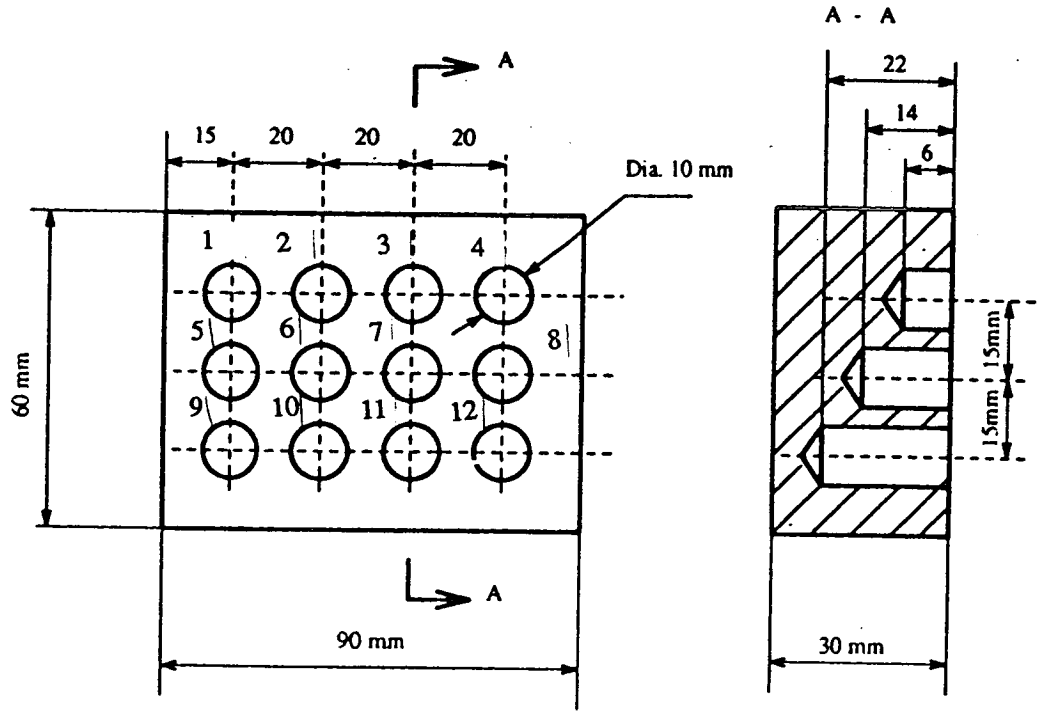


Figure 5.7 Dimensions of Aluminium Mandrel for Hole Throwing Power

No. \ Depth (mm)	0	2	4	6	8	10	12	14	16	18	20	22	24
1	0.16	0.08											
2	0.16	0.08	0.05										
3	0.16	0.09	0.06	0.05									
4	0.16	0.08	0.05	0.04	0.03								
5	0.16	0.09	0.05	0.04	0.03	0.02							
6	0.16	0.09	0.05	0.04	0.03	0.02	0.015						
7	0.16	0.09	0.05	0.04	0.03	0.02	0.015	0.010					
8	0.16	0.09	0.05	0.04	0.03	0.02	0.015	0.010	0.008				
9	0.16	0.09	0.05	0.04	0.03	0.02	0.015	0.010	0.008	0.005			
10	0.16	0.09	0.05	0.04	0.03	0.02	0.015	0.010	0.008	0.005	0.004		
11	0.16	0.09	0.05	0.04	0.03	0.02	0.015	0.010	0.008	0.005	0.004	0.003	
12	0.16	0.09	0.05	0.04	0.03	0.02	0.015	0.010	0.008	0.005	0.004	0.003	0.002

operating condition: current density: 3 A/dm^2 solution: No. 3 bath

Table 5.2 Thickness of Coated Nickel for D.C. Power Supply

0.5 mm PVC plate with 12 holes was covered on the top of surface

No. \ Depth (mm)	0	2	4	6	8	10	12	14	16	18	20	22	24
1	0.35	0.30											
2	0.35	0.23	0.16										
3	0.35	0.23	0.14	0.07									
4	0.35	0.23	0.14	0.08	0.05								
5	0.35	0.23	0.14	0.07	0.05	0.04							
6	0.35	0.22	0.14	0.07	0.05	0.04	0.03						
7	0.35	0.22	0.15	0.08	0.05	0.04	0.03	0.02					
8	0.35	0.22	0.14	0.07	0.05	0.04	0.03	0.02	0.015				
9	0.35	0.22	0.46	0.07	0.05	0.04	0.03	0.02	0.015	0.010			
10	0.35	0.22	0.14	0.07	0.05	0.04	0.03	0.02	0.015	0.010	0.008		
11	0.35	0.22	0.14	0.07	0.05	0.04	0.03	0.02	0.015	0.010	0.008	0.006	
12	0.35	0.22	0.15	0.07	0.05	0.04	0.03	0.02	0.015	0.010	0.008	0.006	0.005

operating condition: current density: 3 A/dm^2 solution: No.3 bath

Table 5.3 Thickness of Coated Nickel for D.C Power Supply with Insulating Plate

No. \ Depth (mm)	0	2	4	6	8	10	12	14	16	18	20	22	24
1	0.25	0.20											
2	0.25	0.14	0.11										
3	0.25	0.16	0.12	0.09									
4	0.25	0.12	0.11	0.08	0.06								
5	0.25	0.16	0.13	0.10	0.08	0.06							
6	0.26	0.15	0.12	0.10	0.08	0.06	0.05						
7	0.25	0.14	0.12	0.10	0.08	0.06	0.05	0.04					
8	0.25	0.14	0.11	0.09	0.08	0.06	0.05	0.04	0.03				
9	0.25	0.16	0.12	0.10	0.08	0.06	0.05	0.04	0.03	0.02			
10	0.26	0.16	0.13	0.11	0.09	0.07	0.06	0.05	0.04	0.03	0.02		
11	0.25	0.15	0.12	0.10	0.08	0.07	0.06	0.05	0.04	0.03	0.02	0.015	
12	0.25	0.14	0.12	0.10	0.08	0.07	0.06	0.05	0.04	0.03	0.02	0.015	0.001

operating condition: pulse current density:3 A/dm^2; duty cycle = 50% frequency: 100 kHz

Table 5.4 Thickness of Coated Nickel for Pulse Plating

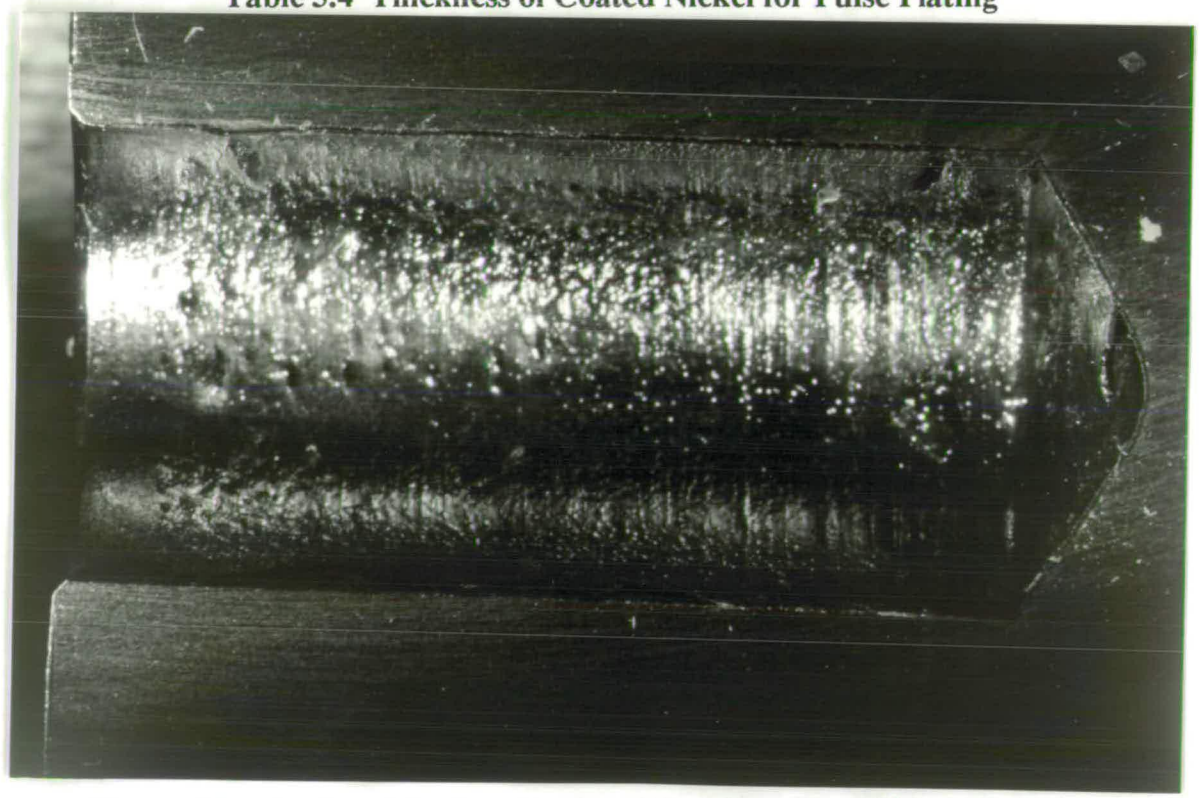


Figure 5.8 Sectional Surface Produced by Electroless Nickel Plating

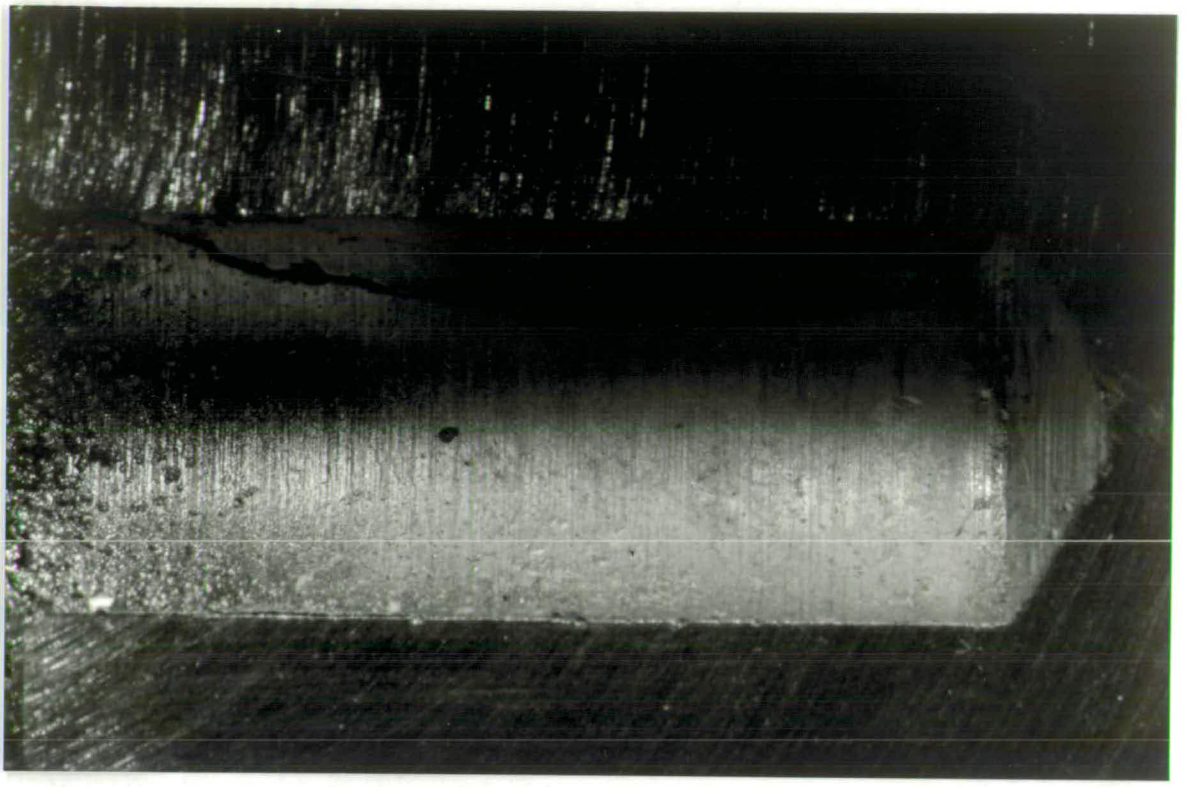


Figure 5.9 Sectional Surface Produced by D.C. Plating

Figure 5.10 Sectional Surface Produced by Pulse Plating

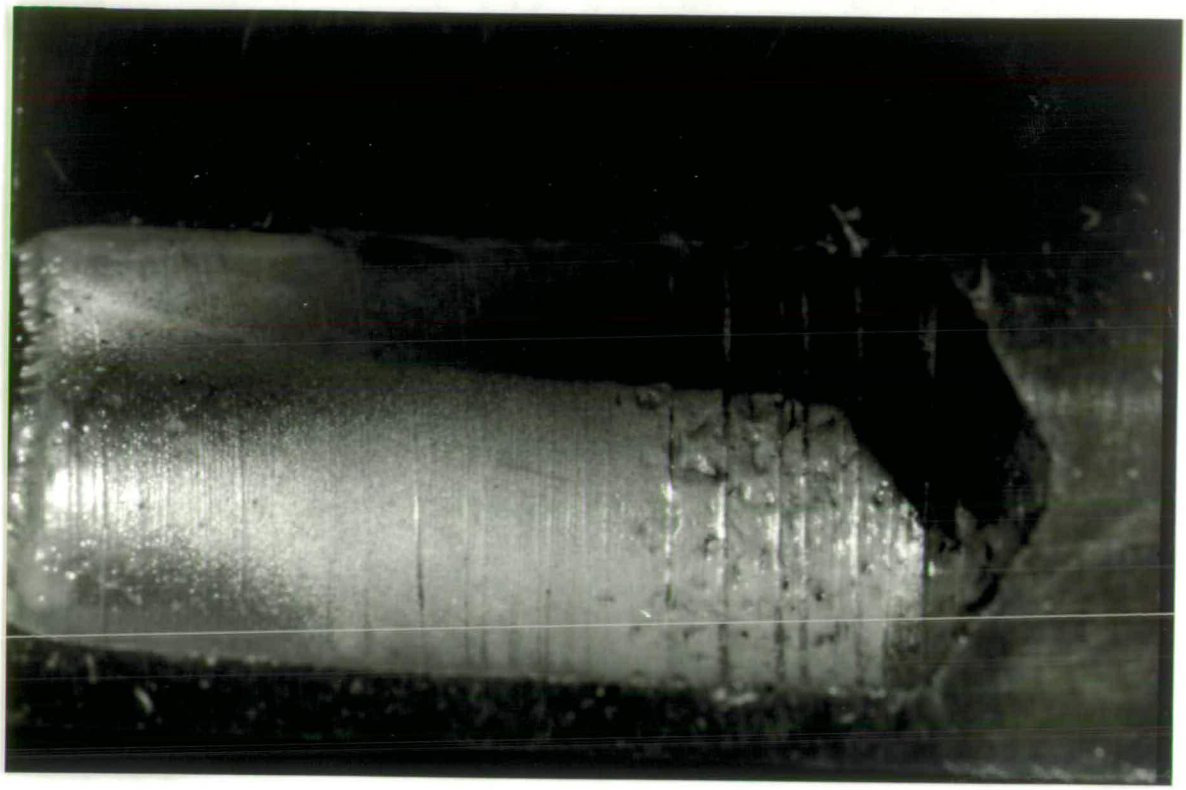


Figure 5.11 Sectional Surface Produced by D.C. Plating with Insulating Plate

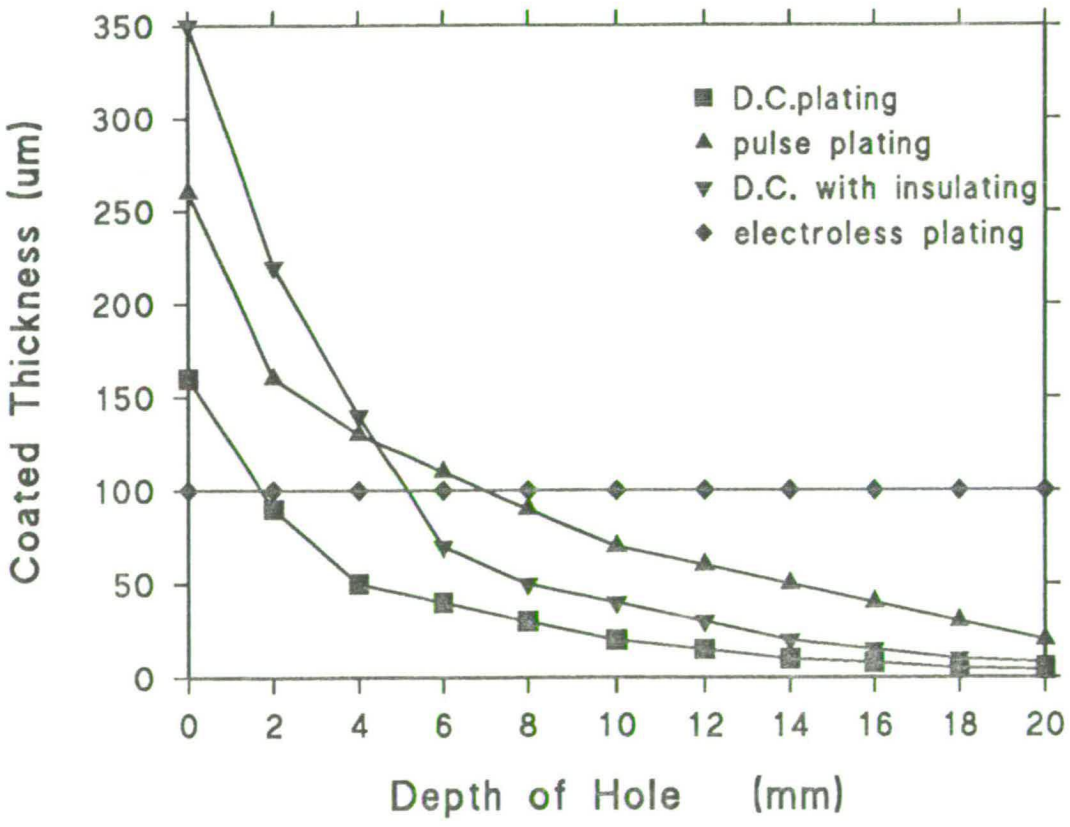


Figure 5.12 Relationship Between the Coated Thickness and Depth of Hole

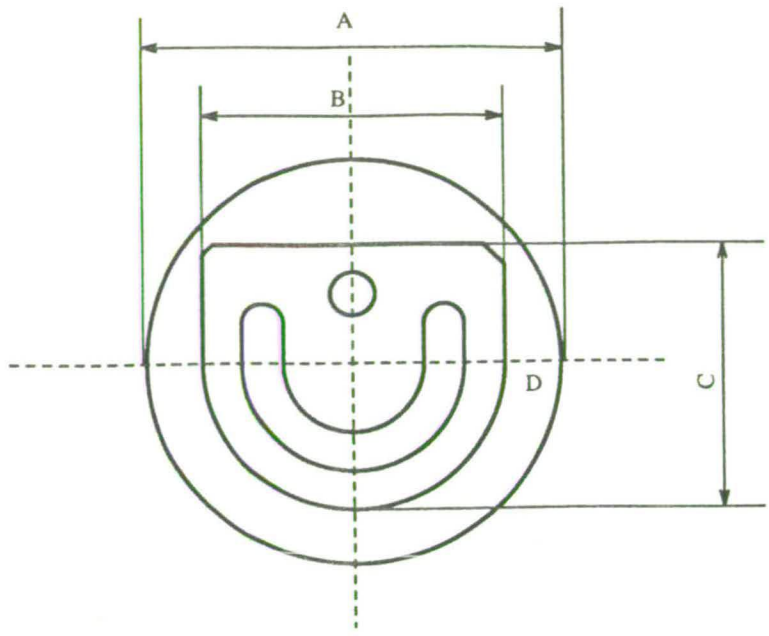
5-4. MEASURING THE ACCURACY OF REPRODUCTION FOR ELECTROFORMING

It is well known that electroforming processes can produce parts with very high dimensional accuracy and reproduce fine surface detail with great accuracy. The following experiment was carried out to determine just how accurately a part can be reproduced.

Two aluminium paper clip mandrels were provided by DTI and are sketched in Figure 5.13, with a photograph in Figure 5.14. Before electroforming, the surface finish of area D was recorded by the Talysurf 4 giving the CLA and chatter marks of the mandrel. After electroforming for 24 hours, the samples were turned in a lathe to remove excess nickel plating so that the shells could be prised carefully off the mandrels. The shells were then measured by micrometer and Talysurf 4 as before.

The Talysurf charts are reproduced in Figures 5.15 and 5.16, The chatter marks of mandrel 1 being given in Figure 5.15a, and of its shell in Figure 5.15b. The same area D(Fig.5.13) was used for each, and it can be seen that the two surfaces are almost indistinguishable. The charts of sample 2 indicate that the surface of nickel shell 2 is better than that of mandrel 2. The difference in peak value between mandrel 2 and nickel shell 2 is about 1.2 μm, and the difference in CLA about 0.25 μm. The results are shown in Table 5.5. The difference in size between the original aluminum mandrels and nickel shells is very small, about 0.01 mm.

It was verified that the electroforming process can provide high accuracy of replica surface and dimension when the aluminum was used for the mandrel.



* A,B,C detail dimensions are shown in Table 1

Figure 5.13 The Dimensions of Paper Clip Pattern

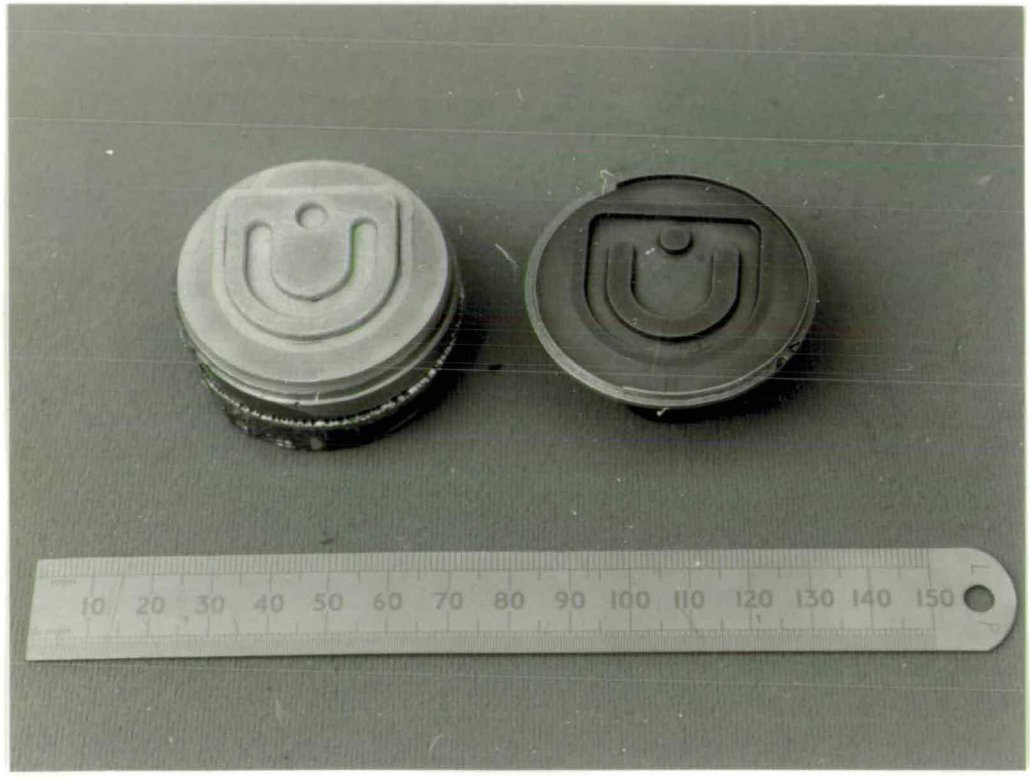
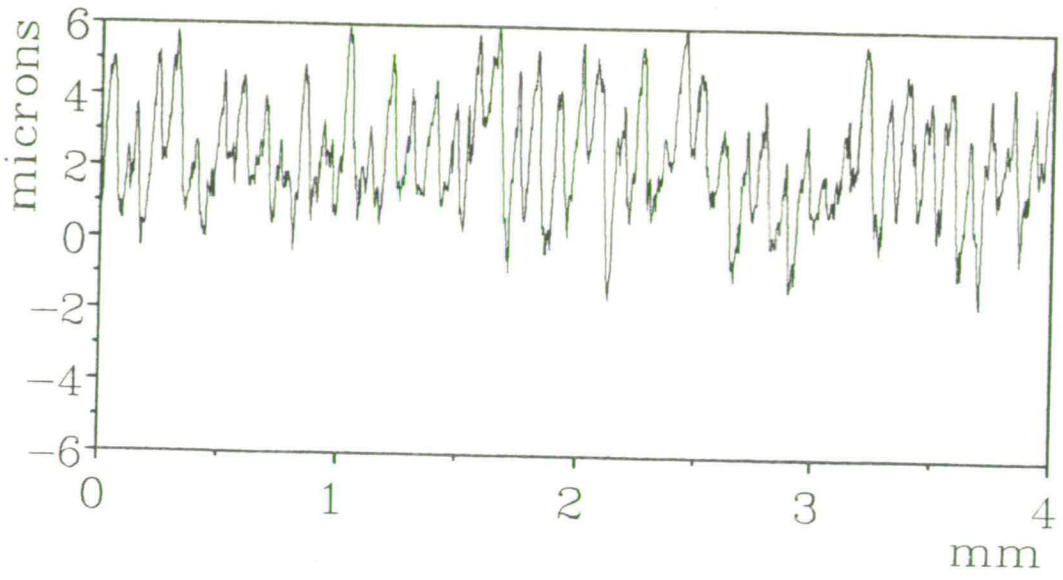
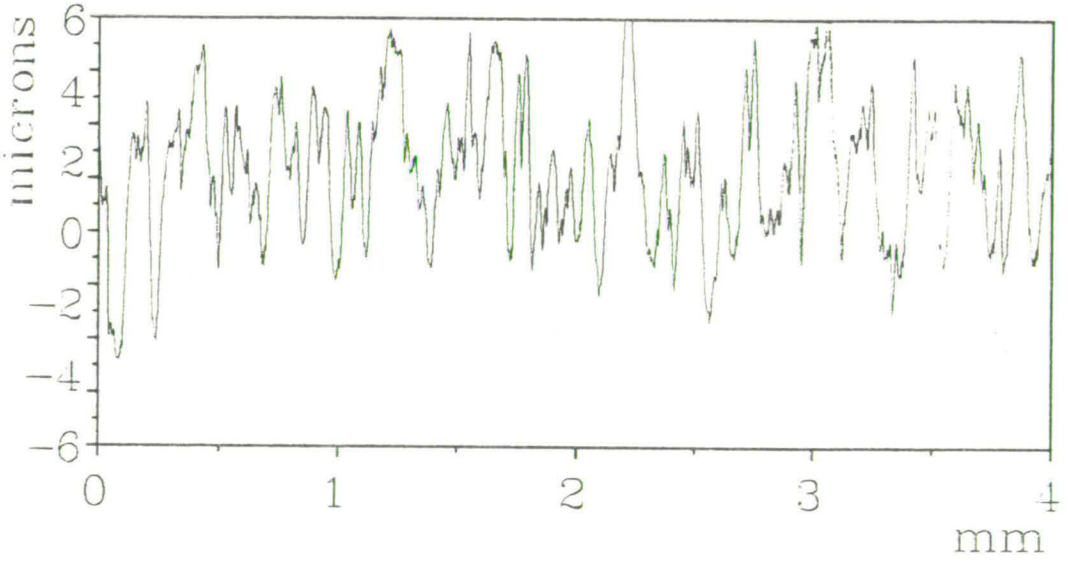


Figure 5.14 The View of Paper Clip Mandrel and Nickel Shell

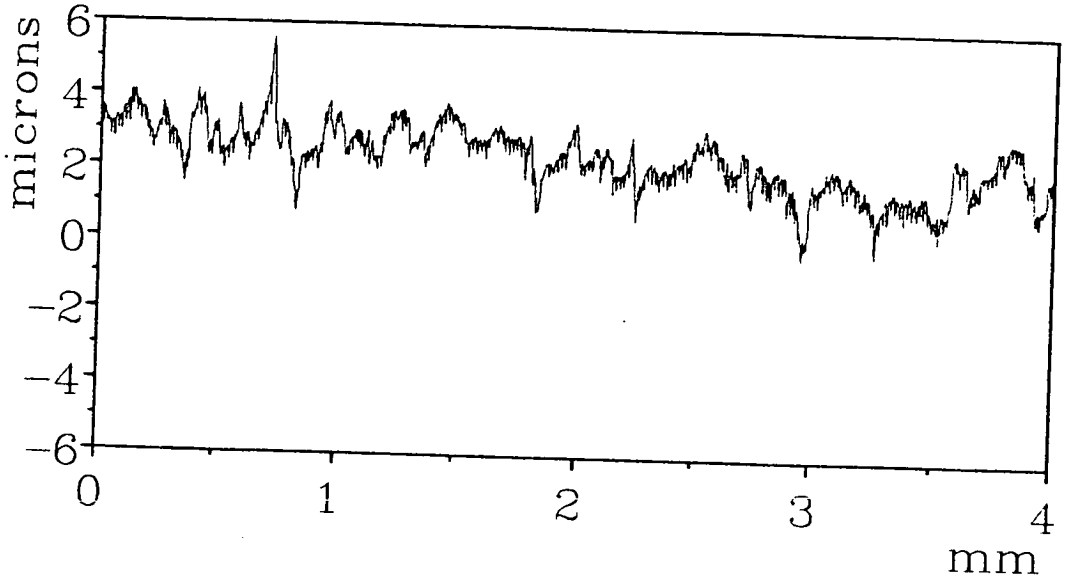


a. Mandrel 1 Chatter Mark

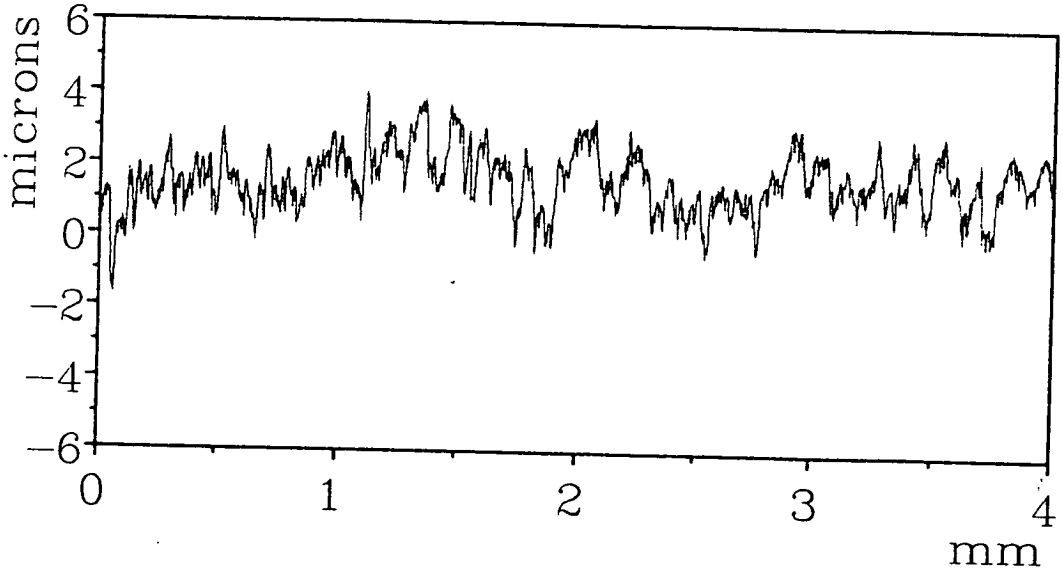


b. Nickel Shell 1 Chatter Mark

Figure 5.15 Talysurf Charts For Mandrel 1 and Nickel Shell 1



a. Mandrel 2 Chatter Mark



b. Nickel Shell 2 Chatter Mark

Figure 5.16 Talysurf Charts For Mandrel 2 and Nickel Shell 2

TABLE 5.5 Accuracy Measurement of Electroforming Process

	Al. Mandrel 1	Nickel Shell 1	Al. Mandrel 2	Nickel Shell 2
A (mm)±0.01	47.01	47.02	47.08	47.09
B (mm)±0.01	29.85	29.86	29.95	29.96
C (mm)±0.01	29.85	29.85	29.95	29.96
CLA (μm)	2.30	2.25	1.45	1.20

CHAPTER 6

INTERFACIAL ANALYSIS AND MOULD TOOL DEVELOPMENT

6-1. INTRODUCTION

Dies and moulds made by conventional methods are costly and have many applications. Engineers are constantly searching for new methods to produce tooling at low cost, with shorter time deliveries, and less tool maintenance without sacrificing quality. A new hybrid electroforming and simultaneous spray peening process (SSP) has been developed to make dies and moulds other than by conventional machining. Lower costs and shorter lead time are possible when this new technique is used.

This process consists of electroforming a nickel shell which is subsequently supported by a backing material, applied by means of a simultaneous spray peening technique. [77] It is essential for successful manufacture of dies and moulds to ensure a strong interfacial bond between the electroformed shell and the

sprayed metal layer. The objective of this chapter is to investigate different bonding techniques and measure subsequent bonding strengths. Several methods of treating the back surface of the nickel shell are described. Finally, the performance of the completed tool is discussed.

6-2. BONDING TECHNIQUES

The hybrid process routes are shown in Figure 6.1 and Figure 6.2. Route A has five steps involved in making tooling where the starting point is a CAD file. The file in this case is used to machine an aluminium mandrel. In route B, a silicone rubber mandrel is used as its starting point. In this case an electroformed shell is produced on the non-conducting mandrel by means of a conductive film of silver applied to its surface. When the electroformed shell is strong enough to be free standing, it is removed from the mandrel and a special casting material, SUPERSTONE, supplied by KERR UK Ltd, replaces the mandrel. This material is quite easy to cast and can endure the high temperature during SSP process. Also it is easy to remove by using hammer to break it. A tool steel backing was then sprayed onto the back surface of the nickel shell and produced a hybrid MUST tool.

No matter which route is chosen, a strong interface is required. In order to obtain a good bond, the electroformed shell must adhere strongly to the SSP backing so that tools applied during injection processes associated with the manufacture of plastics products and aluminium casting, which are the main application areas for this technology. The following methods were tried to improve the adhesion:

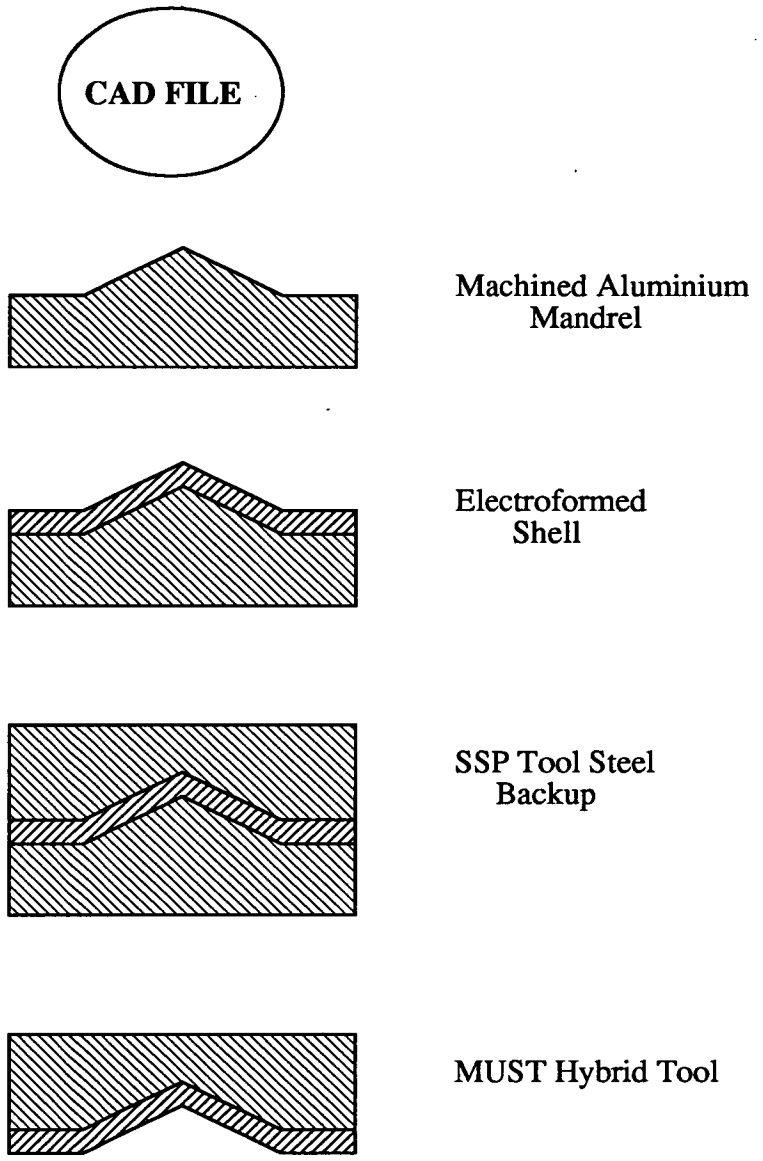


Figure 6.1 The Hybrid Process Routes for Aluminium Mandrel

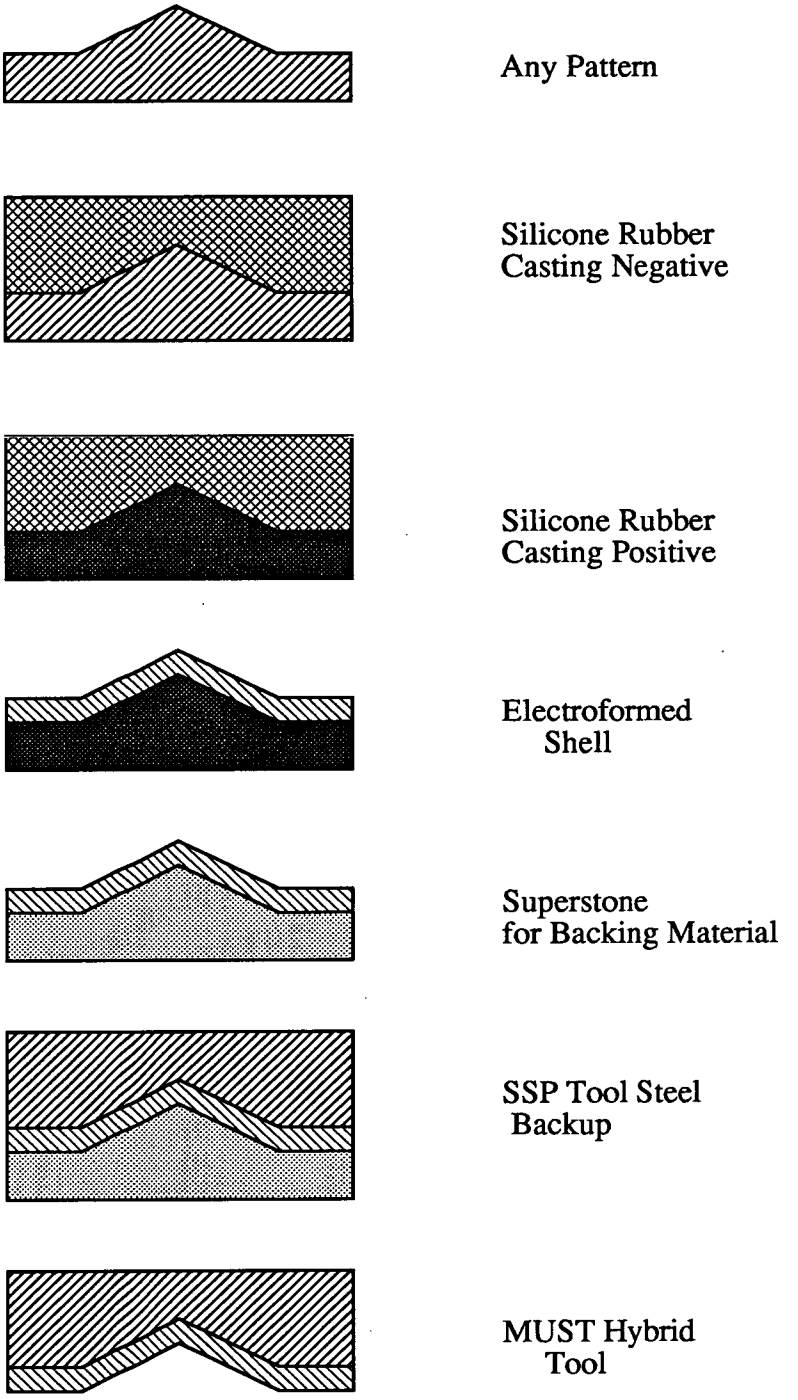


Figure 6.2 The hybrid Process Routes for Silicone Rubber Mandrel

i.Roughening the electroformed shell surface

In this process, the higher current density (30 A/dm^2) was used in last half hour of the electroforming process in order to roughen the back surface of the shell.

ii. Electroformed nickel shell with gold electroplating

After electroforming for 24 hours, the nickel shell was cleaned. The back surface of the nickel shell was coated with thin gold plating of about 20 μm thickness.

iii Combined nickel electro- and electroless forming

With this procedure, the electroless nickel (bath No. 20) was used. A nickel anode and the nickel shell were put into the solution and 3 A/dm^2 current density passed for one hour.

iv. Electroforming and chemical etching

After electroforming for 24 hours, the nickel shell was put in 65% ml/l HNO_3 solution for 60 seconds. The sample was then rinsed in water for ten minutes.

After these treatments, the samples were sent to Sprayforming Developments Limited (SDL) in Swansea to be sprayed with metal. The results showed these methods were not successful. The interface between nickel layer and SSP backing material did not provide adequate adhesion.

An alternative and original procedure, in which electrodischarge machining was used to roughen the surface, provided excellent adhesion at junction of shell and SSP metal. The back surface of the nickel shell was etched by EDM using

matching electrode, which was produced in zinc at SDL. Then, the steel tool material was sprayed onto the nickel surface etched by EDM. Finally, the aluminium mandrel was dissolved in 25% sodium hydroxide.

In this process, it was found that it was quite difficult to make the zinc EDM tool. At the same time, it was very easy to break through the nickel shell when EDM etching, because it was very thin. In order to overcome this problem, a technique called 'pencil' EDM was developed to etch the shell surface. A copper tube was used as an electrode to etch the surface. No matter how complicated the shape of the shell, one copper tube electrode could complete the etching of the whole surface. For example, using the EDM pencil only thirty minutes were required to etch the whole back surface of the motor part shell. The results show that the EDM pencil etch still provides a good bond at the interface. The motor part sample was sectioned and polished. The interface was observed by microscope and a photomicrograph is shown in Figure 6.3. The bond between the nickel shell and SSP backing looks quite strong, even in the corner, without any gap between two layers. The bond strength will be discussed in the next section.

6-3. MEASURING THE BOND STRENGTH BETWEEN THENICKEL SHELL AND SPRAYED METAL

The rapid expansion of adhesive bonding into many applications has called for investigation and testing of bonded joints in many research centres, institutes, laboratories and production plants. A large number of testing techniques used in the testing of metals have been adopted. Semerdjiev[73] introduced some destructive methods for testing bonded metal joints. In general, complex forces are involved in all the basic types of strength testing. A simple tensile test produces shear in addition to tensional stresses, and shear and cleavage tests involve tensional, compressional and shear stresses. In practice, however, in studying the static strength of a bonded metal - to - metal joint the prime interest lies in the normal and the shear stresses. Depending on the type of joint, the peeling load could be of importance. Behaviour under continuous stress and different environmental conditions is also of interest.

In this study, direct tensile testing was used to measure bond strength. In several cases, it is necessary to determine the tensile strength of a bond when the acting forces are applied perpendicularly to the adhesive plane, i.e. when the stresses in the bond line are normal an this direct tensile strength is probably a more accurate measure than other methods. However, there are certain difficulties linked with this test procedure, the main one being the dependence of the tensile value on the geometry of the joint.

Spool-type test specimens were used in this experiment. The measurement of the bond strength was carried out in the Lloyd M30K tensile-testing machine. Views of the clamping and the cross-section of the tensile test specimen are shown in Figure6.4 and Figure 6.5. In this process, 50 mm diameter aluminium

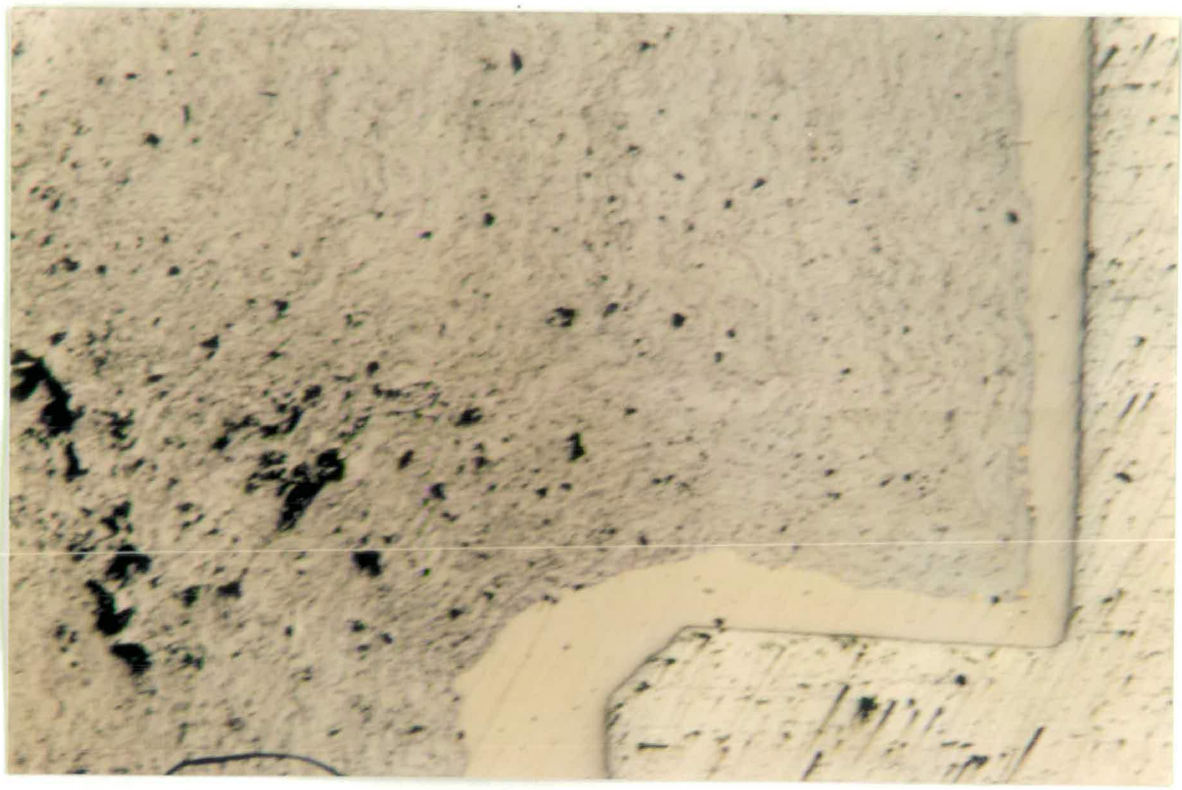


Figure 6.3 The Interface Between Nickel Shell and Sprayed Metal in Motor Part Mould

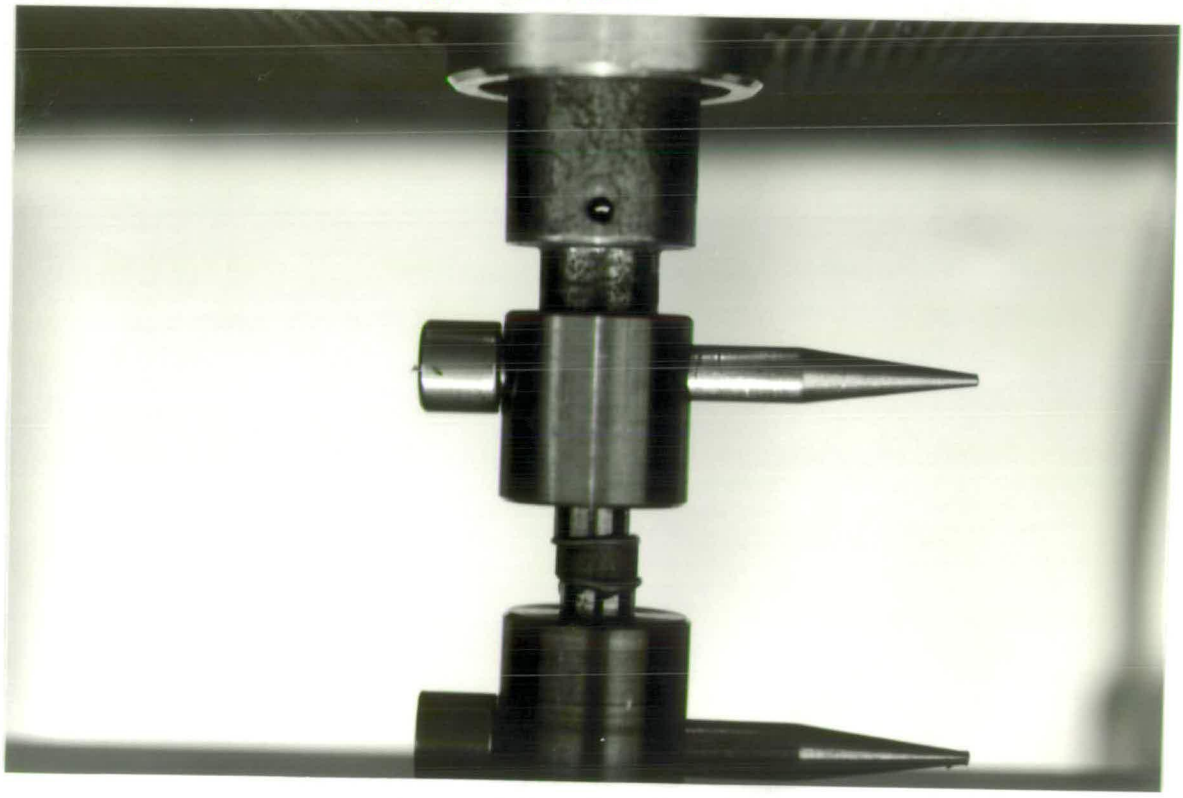


Figure 6.4 View of Bonding Test Clamp

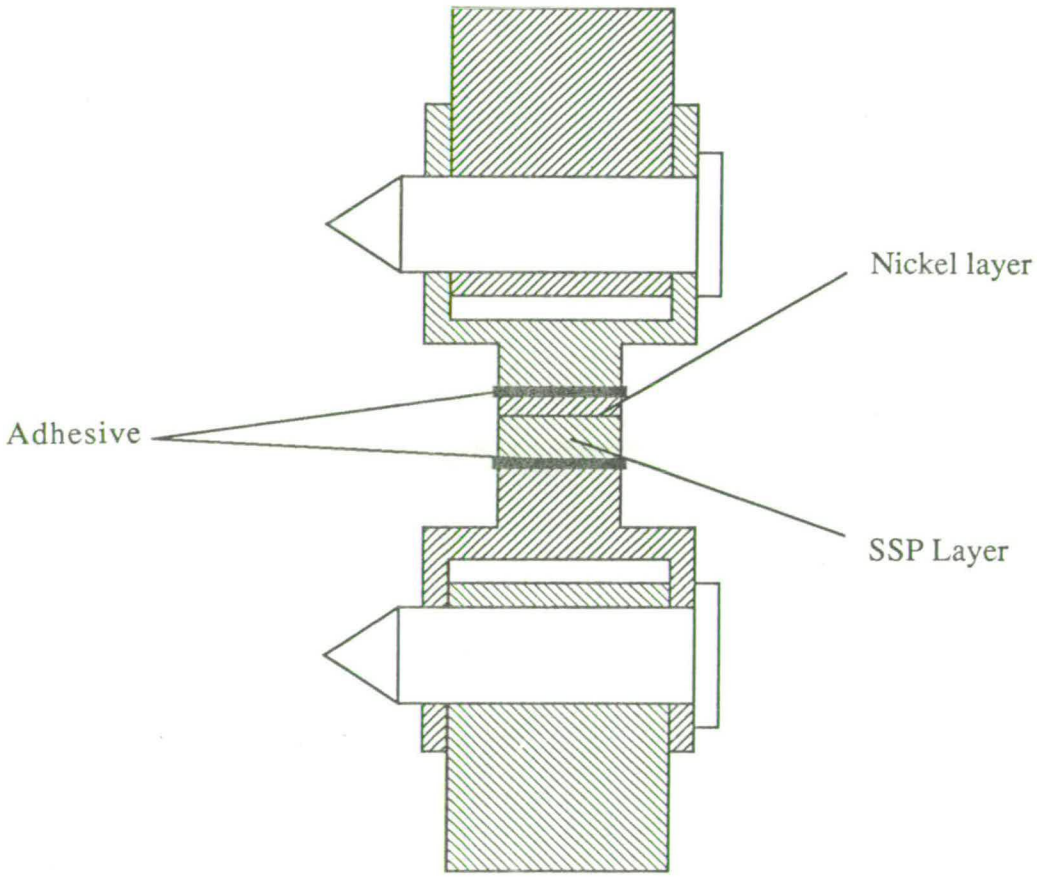


Figure 6.5 Cross - Section of Tensile Sample in Test Grip

discs were used as a mandrel. After treatment, they were electroplated in No. 3 bath for 24 hours. Then the back surface of nickel were etched by EDM at different machining current. Mild steel was sprayed on the back surface by the SSP process. The aluminium mandrels were dissolved in 25% sodium hydroxide. Finally, a copper tube with diameter of 10 mm was used to cut 10 mm diameter cylindrical samples from the specimen by the EDM process. Each disc can make three cylindrical samples. The ends of the samples were ground flat and accurately parallel ready for adhesive bonding to the grips of the tensile testing machining smples. The adhesive, called PERMABOND ESP 110, which was supplied by National Starch & Chemical Ltd, was used to make strong joint between grip and sample. The, surfaces to be jointed must be dry and clean. Chemical treatment of metallic surfaces gives the best strength but abrasion and degreasing is often adequate. Standard tensile tests are used for loading until failure occurs. The tensile strength of the bond is calculated as the failure load divided by the bond area:

$$\sigma_t = \frac{4 \times F}{3.14 \times D^2} \quad (N/mm^2)$$

Where: F = load (N); D: diameter of sample (mm)

During testing, the speed of loading was selected as 6 mm/min. at room temperature. Three similar samples were tested, in each case and the average

value is taken. Table 6-1 shows the results of the bonding strength tests.

It can be seen from Table 6.1 that the bond strength increases slightly with increasing of current in the EDM process. The maximum was 29.8 N/mm^2, etched at 60 A. The back surfaces, which were treated by other methods,(see section6.2) had very low bond strength; even before testing, the nickel shell peeled off the sprayed metal. The reasons why an EDM etched surface produced improved bonding are considered in the next section.

Sample No.	Current of EDM Etched (A)	Bond Strength (N/mm^2)
1	5	24.1
2	10	25.6
3	20	26.3
4	40	28.4
5	60	29.8

Table 6.1 Bond Strength Measurements

6-4. SPARK ERODED SURFACES AND IMPROVED BONDING

Electrodischarge machining (EDM) is one of the major nonconventional machining methods and, briefly, involves the removal of metal by the erosive effects of electrical discharges. Material is removed by a series of discrete discharges that occur in the machining gap between the electrode and the workpiece. The dielectric fluid creates a path for the discharge as the fluid becomes ionized between the two closest points. The initiation of the discharge occurs when sufficient voltage is applied across the machining gap to cause the dielectric to ionize and current to start to flow. The tendency for the discharge to be initiated is increased, when the spacing between the electrode and the workpiece is reduced, the applied voltage is increased, or debris from previous discharges is suspended in the dielectric. The energy of the discharge vaporizes and decomposes the dielectric surrounding the column of electrical conduction.

As conduction continues, the diameter of the discharge column expands and the current increases. The small area in which the discharge occurs is heated to an extremely high temperature, between 8,000 ^{0}C and 12,000 ^{0}C [60] so that a small portion of the workpiece material is elevated above its melting temperature and is removed.

Due to the extremely high temperature in the EDM process, the surfaces discharged by EDM have pock marks, chimneys and resolidified metal droplets. Depending on the input energy and material, craters were about 10 μm to 15 μm deep. The finish or roughness of a surface depends on the current density, the higher the current the rougher the surface.

In order to determine which surface roughness yields the best bond between the nickel layer and SSP backing, five samples were etched at different current , which were 5 A,10 A, 20 A, 40 A and 60 A. Scanning electron micrographs of the different surface roughness are shown in Figure 6.6. The surface roughness increases in distinct steps as the current density increases. Also it can be seen that the pits size on the surface becomes larger with increasing current density. It is believed that these pits enhance the bonding strength.

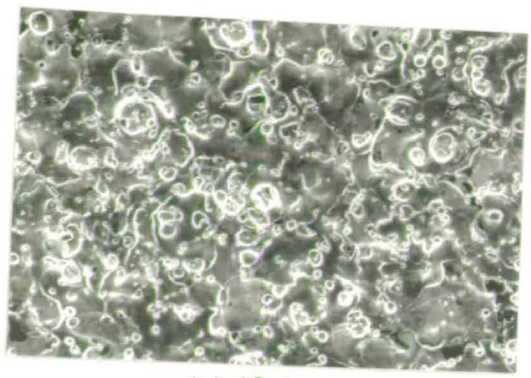
6-5. THERMOPLASTICS INJECTION MOULDINGS

Plastics materials have found a huge variety of application in many areas. These materials offer many desirable characteristics such as interchangeability of parts, excellent finish, desirable electrical and mechanical properties, variety of colour, light weight, thermal insulation, rapid production and low cost. Many of the good properties of the finished product are dependent on the quality of the tool-maker's work. The moulds and dies used are the most important factor in continuous and low-cost production of quality plastics products.

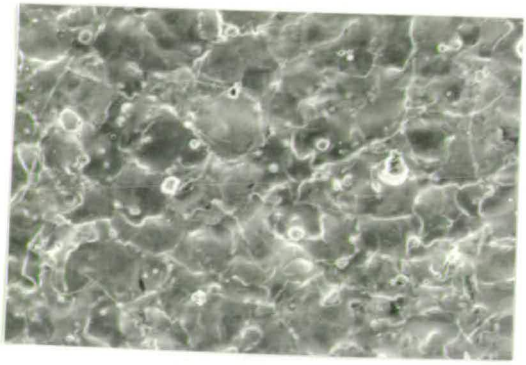
In order to manufacture low cost thermoplastics moulds with short lead times, the MUST process was developed. Based upon a feasibility studycarried out in this project, two moulds, to manufacture a paper clip and a motor part, were made and were used to produce plastic components. The full manufacturing process is detailed as follows:

Firstly, the aluminium mandrel of the motor part and paper clip were supplied by DTI and MAGMA GmbH respectively. (see Figure 5.5 and Figure 5.14) After treatment (see Table 3.2), the aluminium mandrel was coated in No. 10 bath

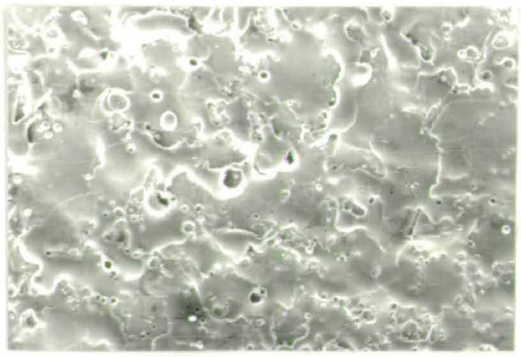
(a) 5 A



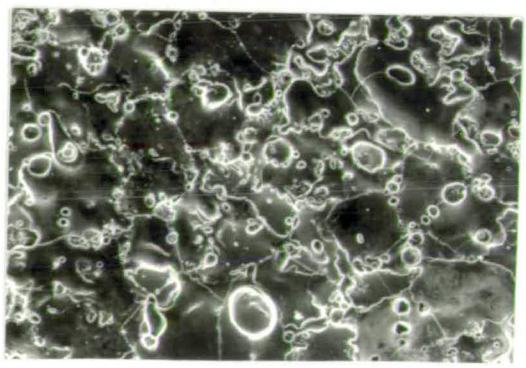
(b) 10 A



(c) 20 A



(d) 40 A



(e) 60 A

Figure 6.6 Scanning Electron Photomicrographs Surface Etched by Different EDM Current (x 50)

at 3 A/dm^2 D.C. current density for 24 hours. Secondly, the back surface of the resulting nickel shell was etched by EDM at 60 A current density. Then thirdly mild steel metal was sprayed on the back surface to support the moulds. Fourthly, the samples were put in 25% sodium hydroxide to dissolve the aluminium mandrel. Finally, these samples were mounted in a plastic injection tool. For the paper clip, four samples, ie. two pairs, were mounted together. The MUST tool can thus produce two components at each shot. The MUST tool was mounted in an ARBURG Injection Moulding Machine, type C270, Max. clamp force of 300 kN in the Danish Technological Institute.

The running of the MUST tool started at 300 bar injection pressure and 150 kN clamping force. The material was polypropylene, called HOSTALEN, initially without colour, later coloured red and then black. The injection pressure and the clamping force were gradually increased until the optimum was reached (injection pressure 550 bar, clamping force 280 kN). At this stage the clips were perfect. The machine was run for 0.5 hour producing 300 perfect paper clips. A close examination of the inserts shown no signs of tool wear.

The same method was used to mount the motor part samples on the MUST tool. With injection pressure 550 bar and clamping force 280 kN, the injection moulding machine ran 0.5 hour and produced 80 plastic motor parts. Then, the MUST tool surface was examined and no tool wear was found. A view of the MUST tool and plastic parts are shown in Figure 6.7 and Figure 6.8.

6-6. HIGH PRESSURE DIE CASTING OF ALUMINIUM

The 'dimple' insert was designed fora high pressure die casting tool. The same method was used to produce the dimple mould, which was mounted on a high pressure die casting machine at MAGMA GmbH. An aluminium alloy was used as the casting material. After the 2nd shot at 650 ^{0}C temperature and 70 MPa pressure, the electroformed nickel shell had cracked and peeled off the backing material, which had been sprayed by SSP. The main reason for the failure of the test is that the nickel shell cannot support such high temperatures and pressures. The results show clearly that MUST hybrid tool cannot be used for high pressure die casting of aluminium.

Figure 6.7 View of Paper Clips Injection Mould

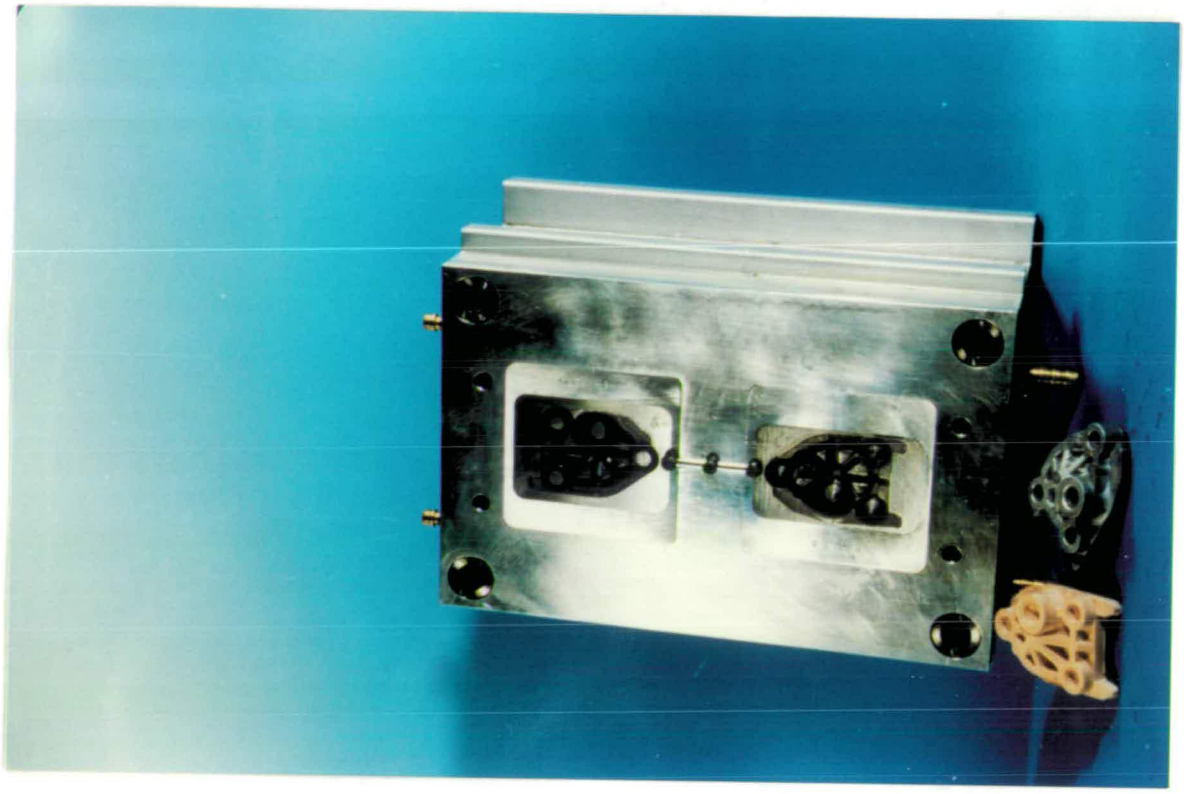


Figure 6.8 View of Motor Part Injection Mould

PART THREE - DISCUSSION

CHAPTER 7

FINAL COMMENTS AND CONCLUSIONS

7-1. FINAL COMMENTS

This feasibility study has shown that the MUST hybrid process has many advantages to the manufacturer of plastic injection moulds. The results show that the mechanical properties of electroformed parts can be changed by the conditions of deposition, such as bath solution, power supply, operating condition and some agents. In order to meet the requirements of industry, most of dies and moulds need to have high quality surface, accurate dimensions, high hardness and high strength. By these researches, very high hardness of 600 HV, over 1,100 MPa ultimate tensile stress and low internal stress (even zero MPa) can be achieved by varying the operating conditions, though not all of these at the same time.

It is well known that most dies and moulds work at high temperatures. For example, the temperature will be up to 650 ^{0}C during high pressure die casting of aluminium. For plastic injection dies and moulds, the temperature is usually over 100 ^{0}C and injection pressure is over 500 bar. The results make clear that

the mechanical properties of the nickel shell are greatly affected by exposure to high temperatures. Ultimate tensile stress, and hardness decrease with the increase of temperature. For example, at room temperature, the nickel shell, which was produced in the hard nickel solution, has about 600 HV and about 1,000 ±20MPa ultimate tensile stress, but after heating at 500 ^{0}C for one hour, the hardness and ultimate tensile stress drop down to about 300 HV and 500±10MPa. The important properties required of materials for metal die casting are resistance to thermal shock and to softening at elevated temperatures. Resistance to softening is required to withstand the erosive action of molten metal under high injection velocity. High die hardness is essential for alloys of high casting temperature, such as, aluminium, magnesium, and copper, which usually necessitates the use of low carbon alloy mould steels prehardened by the manufacturer to maximum hardness consistent with suitable machineability. The typical hardness range is 290 to 370 HV with corresponding tensile strength in the range 875 to 1,240 N/mm^2.[93] However, during casting aluminium, the temperature is usually over 650 ^{0}C so that the mechanical properties of the nickel shell are inadequate so that resulting in creaking the surface of nickel shell, the MUST hybrid tool was damaged.

From Figure 3.10 to Figure 3.13, it can be seen that the hardness and ultimate tensile stress had changed little from room temperature to 200 ^{0}C. This is the reason that the MUST hybrid tool can support the pressure and temperature during plastic injection moulding operations.

With the development of the motor and toy industries, more and more plastic materials are used in the manufacture of products. Therefore, injection dies and moulds play an important part in these fields. Because this new MUST hybrid

process can produce injection dies and moulds at low cost and with short lead times, it will be widely applied in plastic industries in the future.

7-2. CONCLUSIONS

The current research has made a significant contribution to the development of the new technology, the MUST hybrid process. Following conclusions can be drawn from carried out experiments and results achieved in this thesis:

1. The mechanical properties of electroformed nickel can be determined by the composition of the bath and its operating conditions. The highest hardness value 613 (HV) and ultimate strength 1,150 MPa can be obtained in No. 17 bath with 3 ml/l EPC 30 agent.(see page 43)

2. Cobalt sulphate can improve the mechanical properties of electroformed nickel. When No. 10 bath with 5 g/l cobalt sulphate was used as the plating solution, the hardness of the nickel shell can reach up to 357.2 HV, and ultimate strength increased up to 900 MPa.

3. The mechanical properties of electroformed nickel can be greatly affected by high temperature. The hardness value and ultimate strength can drop to about half value.

4. The internal stress can be obtained by selection of operating parameters. No. 1 bath without nickel chloride can produce a low internal stress nickel shell.

5. An electroless nickel layer is not suitable for the surface of dies and moulds, because the layer is very brittle and cannot support the injection pressure from casting aluminium and plastics.

6. Pulse plating can improve the characteristics of nickel deposit. Optimum pulse parameters can provide high throwing power, uniform nickel coating layer and low internal stress. Also it can enhance the hardness, the ultimate strength and decrease the surface roughness of coated nickel with increase of pulse frequency.

7. Electrodischarge etching of the nickel shell surface greatly increases its bond strength to the metal sprayed by SSP. This novel operation seems essential for the MUST hybrid process.

8. The dies and moulds made by the MUST hybrid process are suitable for use as plastic injection moulds, but not for high pressure die casting of aluminium, because the nickel layer on the moulds surfaces cannot support the high temperature and pressure during die casting.

7-3. RECOMMENDATION FOR FURTHER WORK

The two main requirements which create difficulty in the electroforming of shells are:

i). the need to provide uniform thickness all over.

ii). the need to provide very high hardness throughout the life of the mould.

The first concerns throwing power; means of improving this should be developed. In addition conforming anodes, conducting but unconnected metal inserts for cavities, and insulating "screens" should be investigated. Also it is suggested that the electroforming equipment should be designed to rotate the mandrel dur-

ing operation. This method may improve throwing and obtain more uniform thickness of the nickel shell.

High hardness is difficult to achieve and maintain throughout the life of the tool. Alternate thin layers of very high and medium hardness to provide a laminated material and composite deposits of metal with ceramic particles should be investigated. In addition, the possible use of combined electro-and electroless nickel plating with a high nickel, low hypophosphite bath should be assessed since this might provide hard enough deposits but with some ductility.

Concern should be had for commercial implications particularly by seeking industrial support.

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TOOLS MADE BY ELECTROFORMING AND SIMULTANEOUS SPRAY PEENING

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1.ABSTRACT

This paper introduces a novel method of manufacturing tools for die casting, plastics moulding or deep drawing at greatly reduced cost and delivery time compared with conventional procedures. It is being developed as a cooperative venture by five European organisations with support from the EEC.(BRITE-EURAM CONTRACT No. BREU-00429; project No. BE-4621-MUST)

2. INTRODUCTION

Conventional tool manufacture involves the purchase of expensive tool steel, annealing, machining, hardening, tempering, stabilising, grinding, stabilising and perhaps regrinding. Lead times of six to twelve months and prices of the order of £50,000 are not uncommon. Alterations to an existing tool to accommodate desirable design changes are rarely practicable because of the difficulty of machining the hardened metal or of joining parts to it. Accordingly tools are normally required to meet demands for long stable runs of components and this may hinder the introduction of improvements.

Clearly a procedure which would provide tools cheaply and quickly would be advantageous and would permit their economical use for much shorter runs. Such a process 'Manufacture Using Spray Peening Technology'(MUST) is currently being developed by Sprayforming Developments Ltd.; Edinburgh University; Danish Technological Institute; Grundfos International a/s. and Magma G.m.b.H. and consists of electroplating a master to repiicate its surface, simultaneous spray peening to build up a substantial body, and mounting in an appropriate manner.

3. MANUFACTURING PROCESS

1.Mandrel preparation.
2.Coating with hard metal (optional).
3. Simultaneous spray peening.
4. Backing for support.
5. Fitting into a bolster.
6. Testing.

3-1. MANDREL PREPARATION

The mandrel or former may be manufactured by conventional engineering machining procedures. Clearly it should be easily removable from the processed tool. In general, even though this is probably the most expensive part of the process, it is not particularly important that the mandrel be recoverable for reuse since its product may be utilised subsequently or the tool life is adequate. It is important however that the mandrel can withstand the subsequent high temperatures and that it can be processed so that the initial hard metal coating will adhere strongly to it and accurately match its surface contours.

The most convenient mandrel material at present is aluminium since it easily machined, is readily processed for metal coating, has excellent thermal conductivity to carry off the heat released during the subsequent spray peening operation, has a high enough melting point, is inexpensive and readily available in a variety of forms, is easily removed without damage to the partly finished tool.

Other materials have been considered such as optically cured plastics, graphite blocks, but if the hard metal coating is applied from a liquid it is important to avoid a porous material such as Plaster of Paris unless it were impregnated with resin. Any low temperature material such as plastics would need to be removed prior to hot processing and substituted with an accurately conforming and high temperature replacement.

3-2. COATING WITH HARD METAL

Although the metal spraying operation provides a very good reproduction of the mandrel's surface it cannot provide the finest detail. Accordingly electro or electroless plating is used to provide the initial layer or "shell" on the mandrel. Peening can reproduce the finest detail remarkably well but has various difficulties. Hard plating deposits are generally highly stressed and tend to break up unless supported by strong adhesion to the mandrel. In general it is difficult to achieve adequate adhesion to plastics but for metals the conventional preparation procedures developed by the plating industry are adequate. Aluminium is more difficult than some but a double 'zincating' operation replaces the recalcitrant oxide film with a thin layer of active metallic zinc which is a good base for subsequent hard layers. The coating should be hard to minimise indentation, wear resistant and corrosion resistant.

Electroplating has the advantage of being a long established procedure, and where it is used to used provide a self supporting shape rather than a protective coating the term 'electroforming' is used.[1,2,3] Various coatings can be used, chromium and hard nickel being most suitable, and they can be made thick enough to be self supporting when the mandrel is removed. A major problem with electroplating is 'throwing power' and especially with the electrodeposition of chromium. Throwing power is the ability to deposit uniformly over an irregular surface. With deep holes with an aspect ratio greater than one, chromium is often unsuitable. Nickel plating is better and can match the 'throwing power' of the simultaneous spray peening. Electroplated deposits tend to develop irregular growth beyond thicknesses of about 125 microns and produce very rough modular deposits.

The mandrel-to-plating adhesion is not important provided the plating replicates the pattern. For aluminium conventional preparation procedures give a strong bond but this is not a difficulty since it is removed by chemical dissolution. On polymers the adhesion is low and the mandrel can be prepared in such a way that it can be extracted and used again.

Electroless or chemical, deposition here is essentially nickel-phosphorus alloy plating. It is hard: 600 V.H. is deposited and can be heat treated to over 1,000 V.H.N. Its prime advantage is its almost infinite throwing power - the deposit has uniform thickness wherever there is free access of the plating solution. Although ideal in other respects it is extremely brittle especially in thick layers and cannot always withstand the spray-peening operation. Coatings up to 400 microns have been used for electro and electroless shell formation. Adhesion between the shell and the spray deposit is vital for a successful tool and a great variety of etching and overcoating processes were tried before one was established. (Throwing power depends on the composition of the plating solution - resistivity, deposition potential, valency of the metal being important factors. Cyanide solutions e.g. silver, have very good throwing power, chromium with a high valency of six is very poor)

3-3 SIMULTANEOUS SPRAY PEENING

When spraying, the molten steel not only solidifies giving out latent heat and shrinking, but shrinks during cooling and during solid state phase changes. These shrinkage occur rapidly and in a non-uniform manner introducing dimensional changes and internal stresses which have made spraying unsuitable for thick hard deposits. However it has been found that these stresses are removed by suitably shot peening at the same time as spraying hence the name 'Simultaneous Spray Peening' (SPP).[4,5,6]

The tensile thermal stresses, which normally lead to distortion of spray formed deposits, can be neutralised by the compression stresses which are built up by SSP. By adjusting the kinetic energy of the incident shot, distortion of high temperature deposits can be eliminated, enabling precision replication to be achieved in a similar manner to that already used in industrial production with low temperature metals using conventional spray deposition techniques.

A second important effect is the continuous densification of the high temperature deposit by the mechanical work from the impact of the balls.

Factors influencing the quality of the deposit include:
1). Metal deposition rate.
2). Atomisation pressure.
3). Peening velocity and diameter of shot.
4). Peening rate.

The first two affect the deposit mainly because they affect the temperature of the droplets of metal at impact. Increased deposition rate results in higher temperatures and greater shrinkage stresses, and the higher atomisation pressure results in lower temperature with the opposite effect. Increased peening energy either in the form of velocity or rate results in increased compressive effects.

Thus the tensile stresses due to thermal shrinkage can be balanced by compressive stresses from the simultaneous peening operation to provide a very low resultant stress.

The microstructure of the deposit is complex due to the combination of rapid cooling and mechanical work. Porosity is low. The initial layer on a cold mandrel will be martensitic but with continued spraying the average temperature rises to about 400 ^{0}C so that the deposit forms a tempered structure similar to bainite because of its rapid passage through the phase change from austenite. If the mandrel is preheated to above 200 ^{0}C, the martensitic phase will not occur.

The shell produces by the procedure described in section 3-2 is submitted to S.S.P. until an adequate thickness has been formed. One metallurgical problem being investigated is that the initially deposited hard layer may be softened by the heat from subsequent deposition. S.S.P. equipment is shown in Figure 1. A schematic of the S.S.P. equipment gave in Figure 2. A replica of a coin shown in Figure 3.

Figure 1 is a photograph of the S.S.P. equipment, and Figure 2 is a schematic drawing. It consists of a strong square box with ports to carry the active parts. The roof accommodates two arc

netal spraying guns with twin wire feed and a centrifugal nechanically driven shot peener. The front port carries a manipuator, which supports the workpiece, an electric motor for rotation nd enables X-Y motion. The workpiece can also be tilted to 900. At the base exhaust gases and used shot are removed through conical part.

Figure 3 is prepared directly by S.S.P. The quality is quite high ut a sharper image would be provided with prior electroforming.

-4 BACKING AND 3-5 FITTING INTO A BOLSTER

Sometimes no backing is necessary but the material used epends on the type of operation the tool needs to perform. Cement, low melting point alloys, plastics and accurate machinng of the S.S.P. part have all been used to complete the union with he bolster.

-6 TESTING

Mechanical tests on the materials and assemblies have been arried out during the development phase which is continuing. Iowever the real test of the tool can only be performed under norıal production conditions. In one instance 1,000 aluminium high ressure die castings have been produced without apparent effect n the die.

These have involved conventional mechanical tests such as ardness and tensile testing with special consideration being iven to the bond strength between the electroforming layer and he S.S.P. deposit. No unusual or unexpected results were btained and the quality seems sufficiently high for this type of omponent. Much use has been made of metallographic sectionng to investigate the suitability of the spray procedure (angle, patrn of manipulation, deposition rate, etc.). Tests are continuing.

. CONCLUSIONS

. A novel process for manufacture of tools has been devised.
. It will provide tools with much shorter lead times and lower cost.
. Development is well advanced and is continuing.

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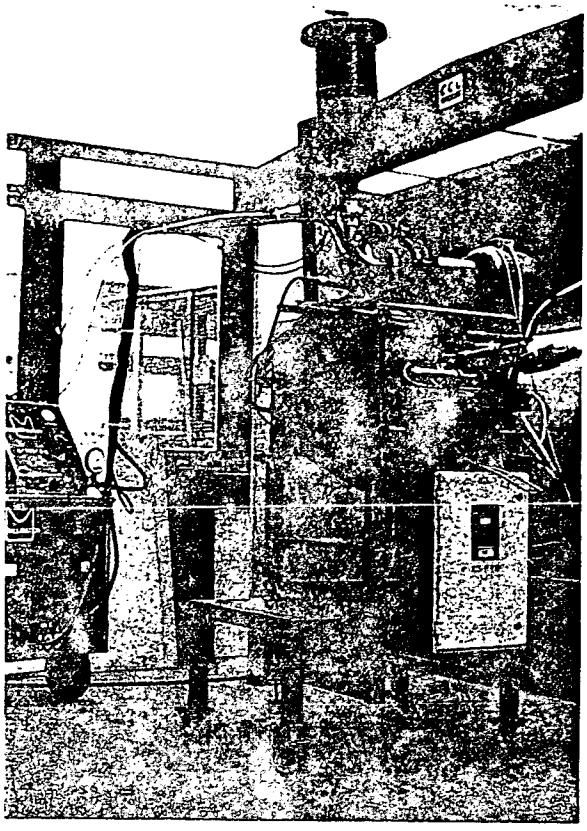

FIGURE 1. S.S.P. EQUIPMENT USED FOR REPLICATION WORK

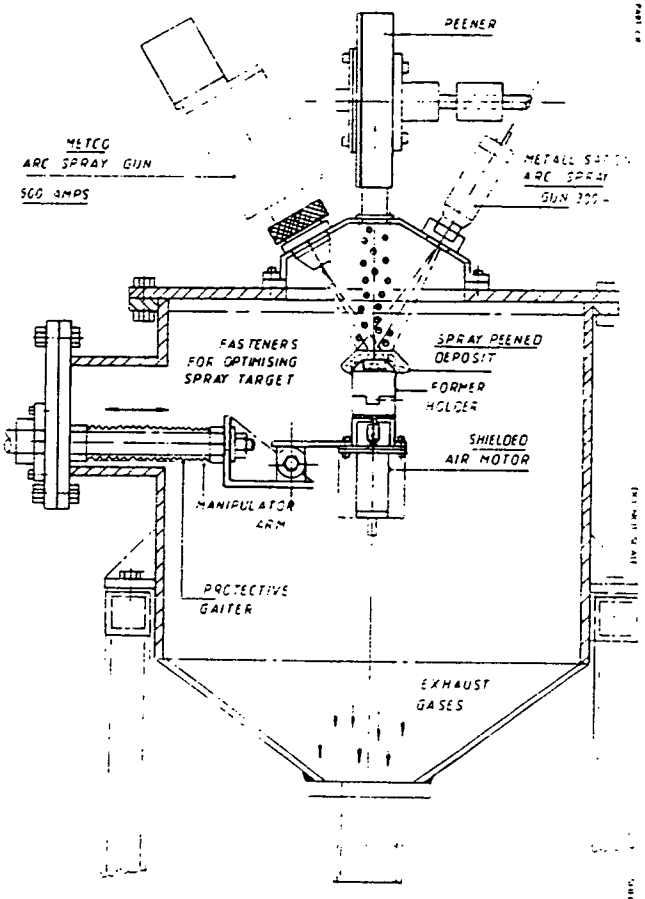

FIGURE 2. SCHEMATIC OF S.S.P. EQUIPMENT

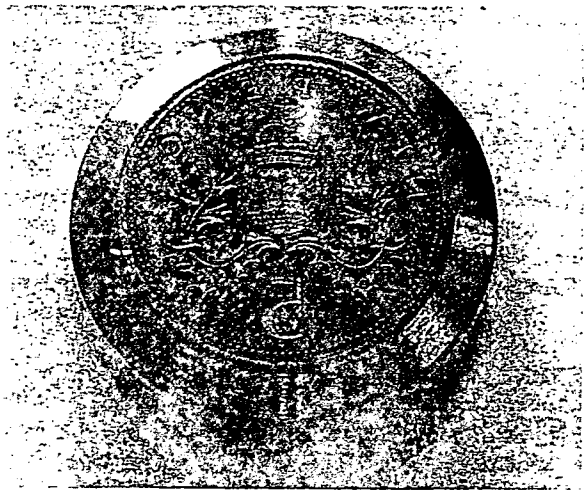

FIGURE 3. S.S.P. TOOL STEEL REPLICA OF FIVE PENCE COIN